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Washington, D. C.



April, 1924

EXPERIMENTAL MILLING AND BAKING  
(INCLUDING CHEMICAL DETERMINATIONS)

By

J. H. SHOLLENBERGER, Grain Supervisor, in Charge Milling Investigations, WALTER K. MARSHALL,  
Baking Technologist, and D. A. COLEMAN, Investigator, in Charge of Research Laboratory,  
Grain Investigations, Bureau of Agricultural Economics.

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An experimental milling and baking laboratory is maintained by the Bureau of Agricultural Economics, Department of Agriculture, in Washington, D. C., for the purpose of testing the milling and baking qualities of wheat, rye, and other grains, and of determining the relationship between these qualities and the physical and other factors which are or may be of use in the grading of grain, particularly in connection with the enforcement of the grain standards act.<sup>1</sup> This laboratory also tests samples of wheat furnished by the Office of Cereal Investigations of the Bureau of Plant Industry, in connection with its study and development of new varieties of wheat, with a view of determining which varieties have superior milling and baking qualities.

The results thus far obtained from tests performed in this laboratory have been of considerable value in fixing the Federal wheat standards and in their interpretation and administration under the grain standards act. The primary purpose of the Federal grain standards, uniform throughout the country and serving as a basis for inspection, market reports, and commercial transactions, is to facilitate trading between buyers and sellers who are operating at some distance apart and who can not meet on common ground with the grain between them for barter. To serve this purpose to the fullest extent, in the case of standards for wheat, the classes and grades must correspond to the relative usefulness of the wheat for milling and baking pur-

<sup>1</sup> A list of the publications issued by the United States Department of Agriculture pertaining to grain standardization is given on page 53.



poses in so far as this is possible in practical application. The value of wheat is almost entirely dependent upon its milling and baking quality, which can be accurately determined only through actual milling and baking tests. Such tests, however, are impracticable for use in grading, because of the time, technique, and equipment required, but are necessary for determining the essential factors in fixing equitable standards. Equitable standards should truly reflect or indicate, first, relative commercial values including differences in quality due to climate or geographical location of production, and second, the relationship existing between the various physical and chemical properties of wheat and its milling and baking quality.

Under the present standards wheat is, first, divided into general types or classes; second, these classes are subdivided into subclasses on the basis of differences in baking strength; and third, the subclasses are subdivided into grades based on cleanliness, quality, and condition.

There are five classes of wheat which take into account botanical differences, including color, texture, habit, and difference in use. For instance, the hard red spring class is composed of the common hard red wheats of spring habit. These are especially well adapted to the manufacture of bread flours. The durum class is composed of all durum wheats. These are used principally for semolina for the manufacture of macaroni and other alimentary pastes. The hard red winter wheat class is composed of the hard red varieties of common wheats of winter habit and is used primarily for the production of bread flours. The soft red winter class is composed of the soft red varieties of common wheats of winter habit and the red club wheats. This class is best adapted to the production of flour for pastry, hot biscuits, and crackers. Some of the wheats of this class also produce a very satisfactory bread flour. The white class is composed of the common white varieties and the white club wheats. With the exception of the white club varieties, which produce a granular, creamy flour, this class is adapted to the same uses as the soft red winter class, and, in addition, on account of its color, is used extensively for the manufacture of those breakfast foods in which the whole kernel is utilized.

Each class of wheat is divided into subclasses which are intended to reflect differences in quality within the class. For example, hard red spring wheat is divided into three subclasses—dark northern spring; northern spring, and red spring. The determining factors are kernel texture, variety, and geographic location of production. In the hard red spring, durum, and white wheat classes, the subclasses are based partly on kernel texture and partly on variety. In the hard red winter class, kernel texture alone determines the subclass, while in the soft red winter wheat class, variety and geographical location of production are the subclass determinants.

The subdivision of the various subclasses into numerical grades is made in accordance with certain definite limitations or requirements as to test weight per bushel, moisture content, total percentage of damaged kernels, heat-damaged kernels, total percentage of foreign material other than dockage, matter other than cereal grains, total amount of wheats of other classes, wheats of special classes, foreign odors, temperature, presence of live insects injurious to stored grain, distinctly low quality, small inseparable stones and cinders, and special varieties.



Some of the factors which serve as the basis for the grades were chosen because of the existence of a definite relationship to milling quality, some for their relationship to baking quality, others for their relationship to keeping quality in storage and transportation, and still others for their relationship to intrinsic worth. In regard to factors affecting milling and baking quality, the test weight per bushel of a wheat of any given class or subclass is a good index to its flour-yielding capacity; the presence of damaged kernels affects the quality of the flour; and the presence of foreign material may affect either or both flour yield and baking quality. The extent to which milling and baking qualities are affected by damaged kernels or foreign material depends upon the amount present and the type. In the case of damaged wheat the quality is also influenced by the degree or extent to which the individual kernels are damaged.

The basis for the present wheat standards is explained in the preceding paragraphs to show the importance of milling and baking experiments, but it should be remembered that milling and baking tests are necessary not only that proper grain standards may be established for commercial inspection purposes but also that the most suitable kinds of grain may be bred, introduced, and grown.

This bulletin contains descriptions of: (1) The method of handling and analyzing the samples received for milling and baking tests, (2) the experimental mill and its operation, (3) the baking laboratory and the method used in baking, and (4) the equipment and methods of analysis used in determining the chemical constituents of grain and mill products.

#### METHOD OF HANDLING SAMPLES.

Each sample of grain received for a milling and baking test is assigned a laboratory number which, with certain information furnished with the sample, is recorded in a book kept for that purpose. The character of information recorded is indicated on the record sheet shown in Figure 1. This information is later transferred to a laboratory card (see figs. 2 and 3), which follows the sample through the various tests applied and forms the final record of the results of all these tests. The size of the samples obtained usually ranges from 4 to 8 pounds.

After the sample has been recorded, it is placed in a tin can, if of the usual size, and in a metal-lined bin if larger. The tin cans are of the baking-powder type, shown in Figure 4, and are of two sizes capable of holding approximately 6 to 8 pounds of grain. The bins are lined with galvanized iron and have a capacity of about 4 bushels. The use of tin cans and metal-lined bins prevents mice and rats, and to some extent insects, from destroying the samples.

In the storeroom, the tin containers are filed or arranged on shelves in numerical order. At frequent intervals the storeroom is fumigated with carbon disulphid to destroy ants, weevils, moths, and other insects which may be present.

#### MECHANICAL ANALYSIS OF SAMPLES.

As the wheat samples are needed for milling purposes they are analyzed for all the factors indicated on the face side of the laboratory card (fig. 2), and their grade determined. In making this analysis,

### Record of Samples Received

[illegible]

Fig. 1.—Record sheet kept for each sample received for milling and baking test. (Size 8½ by 13 inches.)



the grain-grading equipment described in United States Department of Agriculture, Handbook, Form 90, of Official Grain Standards is used.

The mechanical analysis in reality begins as soon as the sample is received at the laboratory, at which time it is examined for odor, temperature, and the presence of live insects. The analysis for the other grading factors namely, dockage, test weight per bushel, moisture, damaged kernels, foreign material other than dockage, smut, kernel texture, and wheats of other classes, is made just preceding the milling test, and if the sample has been in storage for some time before milling, a second examination for odor, temperature, and insects is made at this time. The results of these examinations are entered in their respective places on the laboratory card.

Preparatory to making this analysis two portions are taken from the original sample by use of the Boerner sampling device.<sup>2</sup> One

## WHEAT.

Coupon No. _____		Class <u>Hard Red Spring</u> Place _____		Lab. No. <u>8644</u>	
Submitted by <u>Point of Review</u>		Sec'd at <u>Chicago, Ill.</u>		Sampled <u>Aug. 17, 1922</u>	
Experiments _____		Variety _____		Received _____	
Remarks <u>Duluth composite cargo - 1 sack northern spring</u>					

Basic: Before removal of dockage:		U. S. Grade	Analysis	WHEAT: Other classes—		U. S. Grade	Analysis
Odor: Natural	<u>✓</u>	a	<u>1</u>	<u>H</u>	Hard Red Spring	<u>97.0</u>	
Temperature: Normal	<u>✓</u>	b	<u>1</u>	<u>1</u>	Durum	<u>0.7</u>	<u>0.7</u>
Life weevil and/or	<u>✓</u>	c	<u>1</u>	<u>1</u>	Hard Red Winter	<u>0.4</u>	
Garlic bulbets	<u>✓</u>	d	<u>1</u>	<u>1</u>	Soft Red Winter	<u>1.4</u>	
Moisture: (a) _____ (b) _____ Ave. _____	<u>0.2</u>	e	<u>1</u>	<u>1</u>	White		
Dockage: Sieves _____ Machine _____	<u>0.2</u>	f	<u>0</u>	<u>1</u>	Total	<u>3.0</u>	<u>0.7</u>
Basic: After removal of dockage:					Subclass—Determining Factors.		
Sent dockage: (a) _____ (b) _____	<u>0</u>	g	<u>1</u>	<u>1</u>	Red Durum		
Distinctly low quality	<u>✓</u>	h	<u>1</u>	<u>1</u>	Western Red		
Weight per bushel:		i	<u>1</u>	<u>1</u>	Humpback		
Before dockage <u>58.4</u> lbs. After dockage <u>58.5</u> lbs.	<u>1</u>	j	<u>1</u>	<u>1</u>	Western White		
Sound wheat	<u>27.9</u>	k	<u>1</u>	<u>1</u>	(C) Grains	<u>2.5</u>	
Total damaged wheat	<u>1.0</u>	l	<u>1</u>	<u>1</u>	Analyst's	<u>2.4. Nov. Spring</u>	<u>0</u>
Other than heat	<u>1.0</u>	m	<u>1</u>	<u>1</u>	U. S. Grade		<u>0</u>
Total F. M. other than dockage	<u>0.4</u>	n	<u>1</u>	<u>1</u>	ANALYSIS OF SAMPLE AS MILLED:		
Cereal grains	<u>1.1</u>	o	<u>1</u>	<u>1</u>	Total foreign material	<u>0.3</u>	<u>0.6</u>
Other than C. G.	<u>0.7</u>	p	<u>1</u>	<u>1</u>	Cereal grains	<u>0.3</u>	<u>0.3</u>
Smut balls (weight in per cent in portion A)	<u>0</u>	q	<u>1</u>	<u>1</u>	Reps	<u>0.3</u>	<u>0.2</u>
(A) Grams _____ Total _____	<u>0</u>	r	<u>1</u>	<u>1</u>	Other than	<u>0.2</u>	<u>0.2</u>
Smut balls _____	<u>0</u>	s	<u>1</u>	<u>1</u>	Corn cobs	<u>0.1</u>	<u>0.1</u>
Dark, hard, and vitreous	<u>24.0</u>	t	<u>1</u>	<u>1</u>	Flour	<u>2.1</u>	<u>2.1</u>
Color and texture		u	<u>1</u>	<u>1</u>	Wheat	<u>2.1</u>	<u>2.1</u>
Hard (not soft and chalky)	<u>✓</u>	v	<u>1</u>	<u>1</u>	Analyst completed (date) <u>Sept. 19, 1922</u>		

FIG. 2.—Laboratory card (face side). (Size, 5 by 8 inches.)

portion, approximately 1,000 grams, is used in determining the amount of dockage present and as a basis for the analysis of other factors used in grading in accordance with ordinary inspection practice. After these analyses are made this portion is combined with the remainder of the sample for use in milling. The other portion, approximately 4 ounces, is put in a glass bottle and filed in racks in the storeroom. This bottled portion is kept as a permanent file record of the sample in its original form. Figure 5 illustrates the type of bottles and racks used for this purpose.

The permanent file samples are identified by slips of paper bearing the laboratory number and placed inside the bottle. The bottles are arranged in numerical order in the racks.

The attention given to the examination of the physical characters or condition of the sample of grain preparatory to making a milling

<sup>2</sup> Described in United States Department of Agriculture, Bulletin 287; A device for sampling grain, seeds, and other material, by E. G. Boerner, 1915.

and baking test and the proper recording of this information is of great importance in determining the extent to which the results of the test may be used in studying the relationship between the milling and baking qualities, the physical condition, and the chemical composition of the grain. The fact that the milling and baking quality of wheat is not dependent upon any one factor, but upon a combination of factors, both physical and chemical, makes necessary a complete analysis of these factors in order to interpret properly the results obtained.

### EXPERIMENTAL MILLING.

Experimental milling, although not entirely comparable with commercial milling, gives results which indicate as truly the relative milling value of different wheats as tests made on a commercial mill. It is a well-known fact that different commercial mills milling the

WHEAT.		STRAIGHT FLOUR.		CHEMICAL ANALYSIS.			
Class <i>Hard Red Spring</i>		Lab. No. <i>8644</i>		WHOLE WHEAT.	STRAIGHT FLOUR.	BRAN.	SHORTS.
				Per cent.	Per cent.	Per cent.	Per cent.
Weight per bushel:		Date baked:	<i>9-15-22</i>				
Unscreened:	<i>58.4</i>	Flour used:	<i>340 gm.</i>				
Screened:	<i>58.7</i>	Water absorption:	<i>26.8%</i>				
Recount:	<i>58.2</i>	Volume of loaf:	<i>2270 cc.</i>	Moisture:	<i>12.0</i>	<i>14.1</i>	
Screenings and scourings:	<i>1.3%</i>	Weight of loaf:	<i>493 gm.</i>	Crude protein (N x 5.7):	<i>12.9</i>	<i>11.0</i>	
Moisture before tempering:	<i>12.0%</i>	Color of bread:	<i>91.0</i>	Glisten—Wet:			
Moisture after tempering:	<i>15.0%</i>	Texture:	<i>89.0</i>	Glisten—Dry:			
Date milled:	<i>9-12-22</i>	Crumb:	<i>creamy gray</i>	Ash:		<i>4.07</i>	
Quantity milled:	<i>1200 gm.</i>	Flavor:		Other extract:			
		Odor:		Addity (% lactic):		<i>1.01</i>	
		Remarks:		Crude protein (N x 5.7):	<i>12.7</i>	<i>11.1</i>	
				Basis, 13.5% moisture:			
				Ash, basis 13.5% moisture:		<i>4.07</i>	
MILL PRODUCTS.		REMARKS: <i>Milled granular</i>					
Straight flour:	<i>70.0%</i>	<i>and natural</i>					
Bran:	<i>17.7</i>	<i>color creamy</i>					
Shorts:	<i>12.7</i>						
Total:	<i>100.4</i>						
Loss in milling:							
Gain in milling:	<i>0.4</i>						
Relative humidity:	<i>70.0%</i>						
Air temperature:	<i>79.0°</i>						

FIG. 3.—Laboratory card (reverse side of fig. 2).

same kind of wheat or different operators at the same mill milling the same kind of wheat, or even the same operator milling the same kind of wheat on the same mill at different times, will produce results which may vary considerably. Tests made on the experimental mill by use of the methods described in this bulletin show less variation in milling yields and in baking quality of flour than some of the variations which have been observed in commercial milling.

Because of the small quantity of wheat, small expense, and short time required for making tests on the experimental mill, it is more practicable than a commercial mill for use in determining the relative milling and baking value of different wheats and in studying the relation of grading factors to milling and baking value. Experimental milling and baking tests are also particularly important in determining milling and baking values of small samples obtained from wheat-breeding experiments.



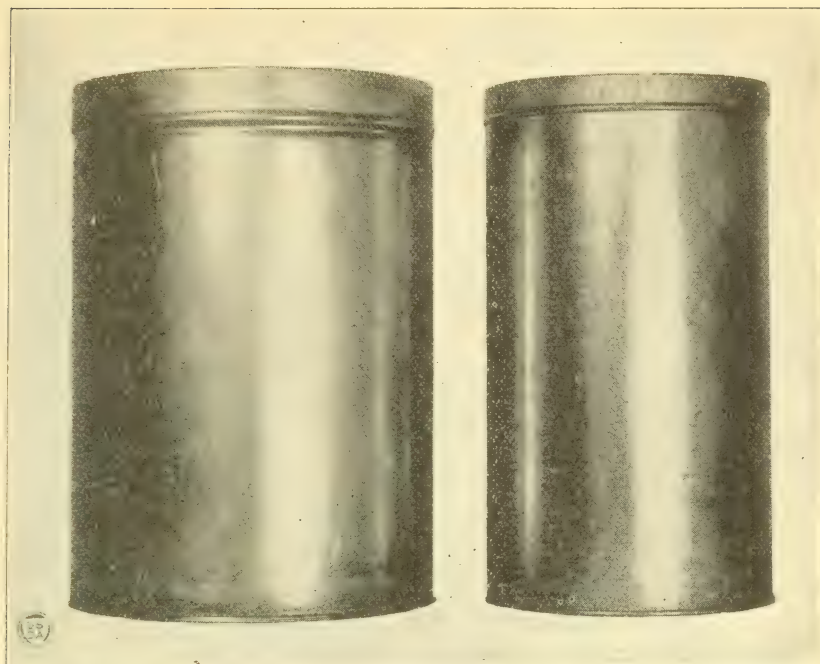


FIG. 4.—Type of containers in which samples weighing from 4 to 8 pounds are stored.

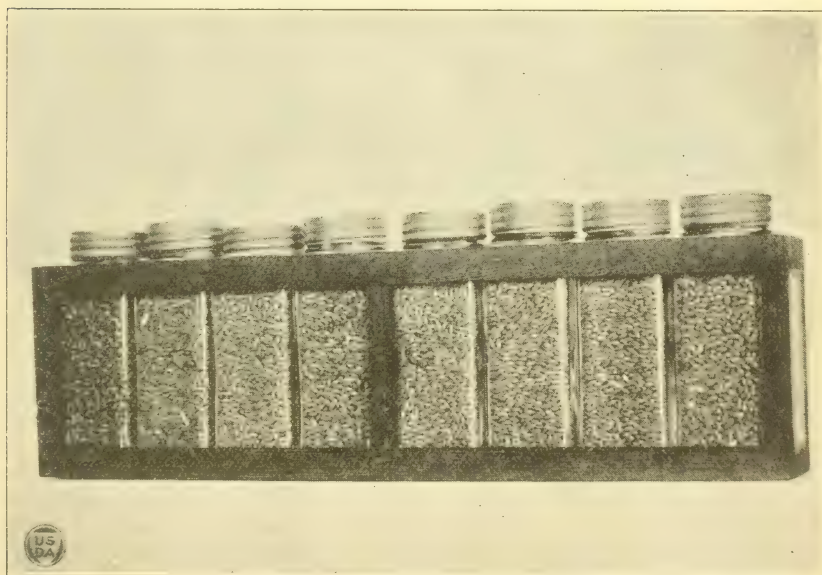


FIG. 5.—Bottles and racks in which a portion of each sample is preserved for future reference.

In addition to furnishing data for use in connection with grain standardization and breeding work, an experimental mill of this type can be used for obtaining milling information valuable to the operative miller. Subjects of this nature, which may be investigated, include the best way to temper or condition various types of wheat, the effect of the fineness of granulation on the baking quality of flour, the effect of various systems of flow on the quantity and quality of the finished product, and the effect of atmospheric humidity on milling efficiency and quality of products.

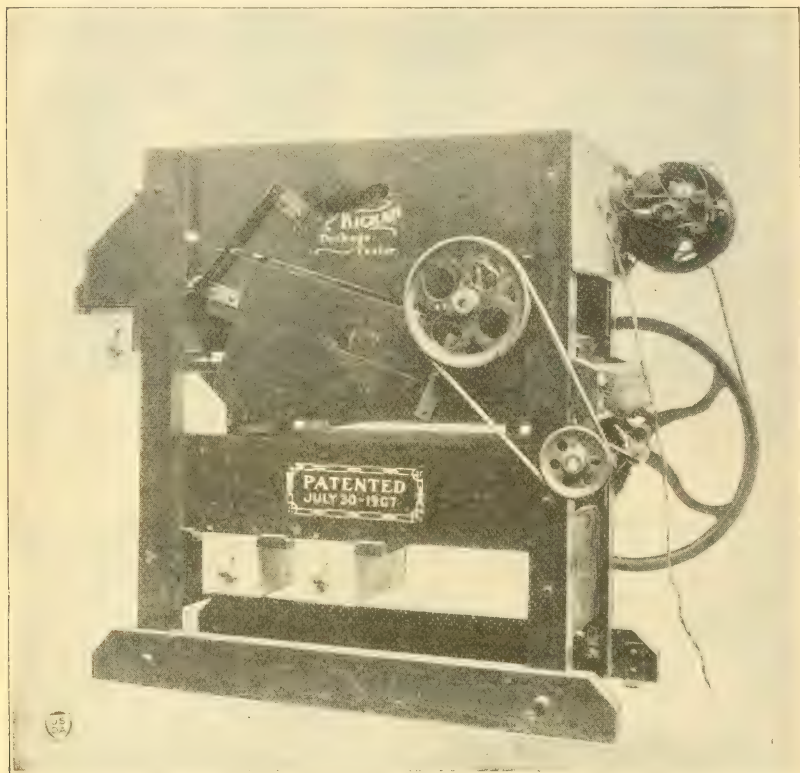


FIG. 6.—“Oat kicker” used in cleaning samples.

#### PREPARATION FOR MILLING.

Before the actual grinding or milling of a sample begins certain preparatory operations must be performed. First, the sample must be cut down to a size appropriate for making the test; this is done on a Boerner sampler. If the sample does not contain more than 5 per cent of foreign material, the size to which it is cut is 1,900 grams. If a considerable amount of foreign material is present, a greater number of grams is taken so that after the screenings and scourings are removed and a 200-gram portion for moisture determination taken out, at least 1,500 grams will remain for milling.



The various operations in the preparation of the sample for milling are as follows:

- (1) Determining the test weight per bushel of the wheat as received.
- (2) Cleaning the grain over an "oat kicker" (fig. 6), and running it through a small milling separator (fig. 7) to remove the foreign material present.

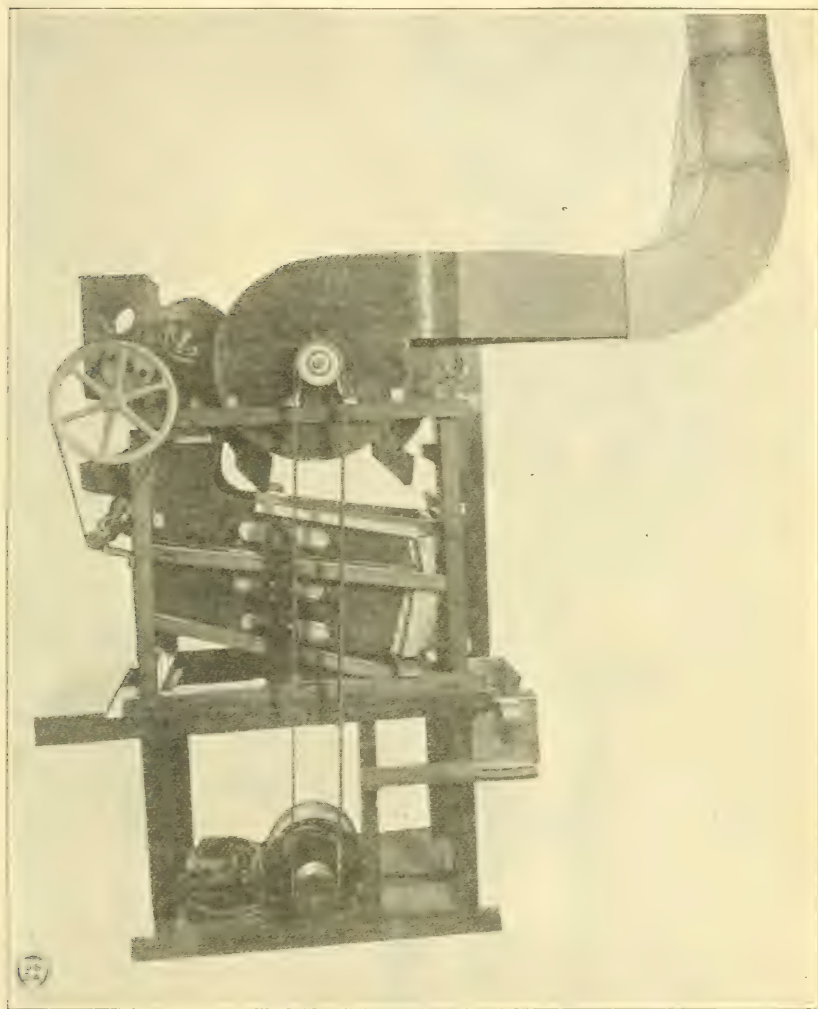


FIG. 7.—Experimental milling separator used for cleaning samples.

- (3) Weighing the cleaned grain. (This weight serves as the basis for determining the amount of foreign material removed.)
- (4) Determining the test weight per bushel of the cleaned grain.
- (5) Scouring the grain in a wheat scourer. (Fig. 8.)
- (6) Weighing the scoured grain.
- (7) Determining the test weight per bushel of the scoured grain.

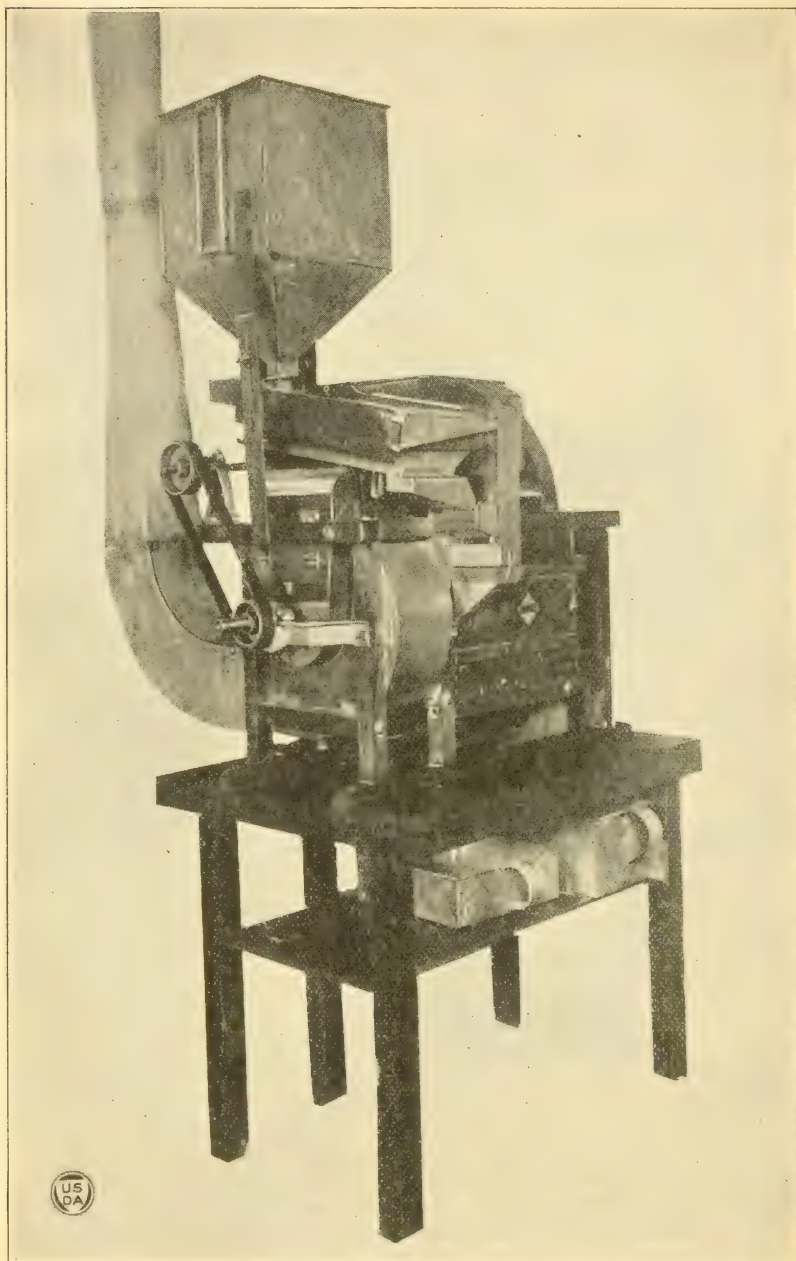


FIG. 8.—Experimental wheat scourer used in cleaning samples.



(8) Determining the moisture content of the grain. (To do this a 200-gram portion is taken from the sample.)

(9) Reducing the scoured sample to exactly 1,500 grams and placing it in a tin can ready for tempering.

(10) Tempering the sample. A complete record of the data obtained in connection with these tests is kept on a milling record sheet, as shown in Figure 9, from which certain of the data are later transferred to the laboratory card. (Fig. 3.)

The tempering process consists of adding sufficient water to the wheat to raise its moisture content to the percentage desired for the milling test. The water is put in the can with the wheat and the latter thoroughly shaken. This is done on the afternoon preceding the day the sample is to be milled. After the water is added and the wheat thoroughly shaken, the can is kept closed until the time of milling.

## MILLING REPORT

Grade and variety <u>Soft Red Winter</u>	Secured at <u>Chicago, Ill.</u>	Kind of grain <u>Wheat</u>	Sample No. <u>48935</u>
Weight per bushel of original sample (uncleaned) <u>58.2</u> lbs.		Quantity milled <u>1500</u> gm.	
Weight before cleaning <u>1800</u> gm.		Date <u>7/27/27</u> Milled by <u>Hager</u>	
Weight after screening <u>1792</u> gm.			
Loss in screening <u>8</u> gm. <u>0.4</u> per cent.			
Weight per bushel after screening (screened) <u>58.5</u> lbs.			
Weight after scouring <u>1759</u> gm.			
Loss in scouring <u>33</u> gm. <u>1.8</u> per cent.			
Loss in cleaning (screenings and scourings) <u>41</u> gm. <u>2.3</u> per cent.			
Weight per bushel as tempered (scoured) <u>58.1</u> lbs.			
Moisture content before tempering <u>10.9</u> per cent.			
Quantity tempered <u>1500</u> gm.			
Water added in tempering <u>54</u> c. c.			
Weight of sample after tempering <u>1554</u> gm.			
Moisture content after tempering <u>14.0</u> per cent.			
Cleaned by <u>E.H.</u>	Tempered by <u>H</u>		
Dry bulb thermometer <u>80</u>	Wet bulb thermometer <u>A.</u>		
Relative humidity <u>45</u>	Readings by <u>A.</u>		
Form M. T. 4.			

MILL PRODUCTS.	
	gm. per cent.
Straight flour	<u>1112</u> <u>74.1</u>
Bran	<u>203</u> <u>13.5</u>
Shorts	<u>168</u> <u>11.2</u>
Total	<u>1483</u> <u>98.8</u>
Loss in milling	<u>17</u> <u>1.2</u>
Gain in milling	
Remarks: <u>Adm. - natural</u> <u>Glenn - white</u> <u>Milled - soft</u>	

FIG. 9.—Milling record sheet. (Size, 5½ by 9⅞ inches.)

The moisture content considered desirable and proper for the experimental milling of soft wheats is 14 per cent and of hard wheats 15 per cent.

With the moisture content of the sample determined, the correct formula for calculating the amount of water to add is as follows: The weight of the sample times the quantity

$$\frac{100 - \text{the per cent of moisture in the wheat}}{100 - \text{the per cent of moisture desired to be in wheat after tempering}} - 1$$

equals the weight of the water to add in terms of the weight units used for the sample. Example: The moisture in a 1,500-gram sample of wheat is 12.2 per cent. How much water should be added to the wheat to raise its moisture content to 15.0 per cent? Solution:

$$1,500 \times \left( \frac{100 - 12.2}{100 - 15.0} - 1 \right) = 49.4 \text{ grams of water, or } 49.4 \text{ cubic centimeters,}$$

since 1 cubic centimeter of water weighs 1 gram.

For the convenience of the miller, a table of equivalents has been prepared which shows the number of cubic centimeters of water to be added to wheats of various moisture contents in tempering them to 14 and 15 per cent, respectively. (See reference, Table 1.)

#### MILLING EQUIPMENT.

The experimental mill installed in the laboratory of the Bureau of Agricultural Economics consists of two units or mills. (See fig. 10). Each unit consists of four stands of 6 by 6 inch rolls, and one box sifter. Three pairs of the rolls of each unit are corrugated and one smooth. Each unit is driven by a 5-horsepower motor of 1,150 revolutions per minute speed. The two sifters, which are mounted together, are driven by a 1-horsepower, slow speed motor.

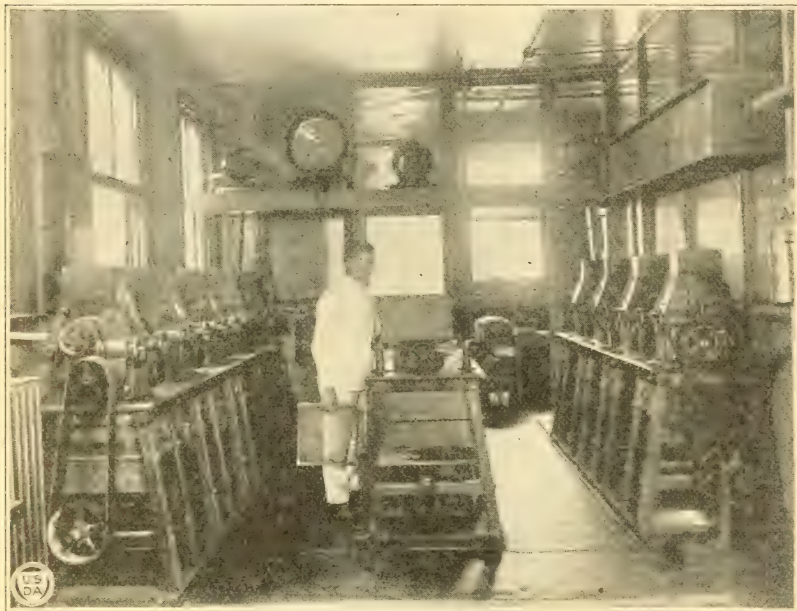


FIG. 10.—Interior of experimental mill.

The milling equipment also includes a humidifier, by means of which the relative humidity of the atmosphere within the mill is maintained at not less than 55 per cent. This is done because the amount of evaporation of the moisture contained in the wheat is affected by differences in relative humidity.<sup>3</sup> Both the relative humidity and the temperature of the air within the mill at the time of grinding are recorded on the milling record sheet.

#### MILLING OPERATION.

The operation of an experimental mill necessarily differs in some respects from that of a commercial mill. In the experimental mill, there is no continuous or automatic flow of stock from one machine to another as in a commercial mill. This is an advantage in that it

<sup>3</sup> United States Department of Agriculture, Bulletin No. 1013: The influence of relative humidity and moisture content of wheat on milling yields and moisture content of flour, by J. H. Shollenberger, 1921.



gives the operator a better opportunity to vary his method of grinding and bolting to suit the character and condition of the individual sample. Furthermore, it decreases the possibility of losing material or of contaminating one sample with another because of the fewer number of places in which material may lodge. Other points of difference are the absence of purifiers and bran and shorts dusters. In spite of these differences, a skillful and experienced operator is able to accomplish results on this mill which compare favorably in quality and efficiency with the work of commercial mills. An approximation of the procedure followed and the sizes of cloths used are given in Figure 11.

To accomplish the five breaks shown on the flow sheet in Figure 11, the first stand of rolls, having 16 corrugations per inch, is used for the first and second breaks, the second stand with 20 corrugations per inch is used for the third break, and the third stand of rolls, having 24 corrugations per inch, is used for the fourth and fifth breaks. In those instances where one stand of rolls is used for two breaks, the rolls are reset when changing from one break to the other to grind to the fineness desired. The speed differential of the break rolls is  $2\frac{1}{2}$  to 1.

The smooth rolls are used for the middlings and tailings reductions. The reduction of the different grades of middlings stock on these rolls is merely a matter of proper adjustment. The speed differential of the smooth rolls is  $1\frac{1}{2}$  to 1.

The sifter is so constructed that five sieves may be used at one time. The sieves used in the sifter in making separations from the first break grinding are, from top to bottom, clothed with No. 16 wire, 30, 50, and 70 grit gauze, and 10 XX silk, respectively. The sieves used for the separation of stock from the second, third, fourth, and fifth breaks are, with the exception of the top sieve, the same as for the first break. The top sieve used for sifting second break is clothed with No. 18 wire, third and fifth breaks with No. 20 wire, and the fourth break with No. 24 wire.

The various grindings necessary for milling a sample and the size of sieves to be used in the sifter after each grinding are indicated on the flow sheet shown in Figure 11.

In milling a sample the ground material is transferred by hand from the rolls to the sifter and from the sifter to the rolls. All the separations resulting from each sifting are not removed immediately from the sifter, but some are left to accumulate through the siftings of several different grindings.

The products—bran, shorts, and flour—obtained from the milling of a sample are weighed, the weights are recorded on the milling record sheet shown as Figure 9, and together with other weights recorded thereon form the basis for calculating percentage milling yields.

A 150-gram portion of the flour is placed in an air-tight can for moisture, ash, nitrogen, and any other chemical determinations desired. The remainder of the flour is placed in a sample can and set aside for the baking test.

This completes the milling of the sample. By following this procedure an operator can mill, depending on the character and condition of the wheat, from four to six 1,500-gram samples in a 7-hour day. The next step in the investigation of the sample is that of baking the flour.





## EXPERIMENTAL BAKING.

In baking bread, two general methods are in common use, namely, the sponge method and the straight dough method. The former method is used in the experimental milling and baking laboratory in connection with investigations for the purpose of the grain standards. The equipment and formula used and the procedure in making tests are described below. From 16 to 18 samples are baked in one day.

The baking formula and method described here have been used in this laboratory since 1915 so that all the baking results should be comparable. This laboratory is now investigating recent advances in experimental baking procedure with a view to making changes in method if found desirable.

## BAKING EQUIPMENT.

- Tin flour pans, size  $6\frac{1}{2}$  by 8 by  $2\frac{1}{2}$  inches with covers. (Fig. 12.)
- Earthen crocks, size  $\frac{1}{2}$  gallon, with tin cover, for sponging and doughing. (Fig. 12.)
- Iron baking pans, size 3 by  $5\frac{1}{2}$  inches at bottom,  $3\frac{3}{4}$  by  $6\frac{1}{2}$  inches at top and  $5\frac{1}{2}$  inches deep. (Fig. 12.)
- Electric agitator for mixing yeast solution. (Fig. 13.)
- Dough mixing machine. (Fig. 14.)
- Proofing cabinet, 2-compartment, 12 upper and 12 lower. Size 1 foot 8 inches by 2 feet 10 inches by 5 feet 1 inch, electrically heated and equipped with an automatic thermo-regulator for controlling the temperature of the air within. (Fig. 15.)
- Double compartment electric oven with snap switch for high, low, and medium heats, and equipped with thermometer for each compartment. (Fig. 16.)
- Loaf measuring device, graduated in cubic centimeters, to show quantity of flaxseed displaced by baked loaf. (Fig. 17.)



FIG. 12.—Tin flour pan, earthen crock, and iron baking pan used in baking.

## BAKING PROCEDURE.

The following formula for baking is used:

	Grams.
Flour.....	340
Sugar.....	15
Salt.....	5
Yeast.....	10
Water (distilled), quantity sufficient to produce a dough of proper consistency.	

The flour samples are usually aged a week before baking. The quantities of flour and salt required in the tests are weighed out the afternoon preceding the day the test is to be made. The flour is

put in the flour pan, the salt in the earthen crock, and placed in the proofing cabinet, until the time of baking.

On the day of baking 150 cubic centimeters of distilled water is put into the crock containing the salt and agitated until the salt dissolves. The 10-gram portion of yeast required for the test is thoroughly mixed by means of an electric agitator with 30 cubic centimeters of lukewarm distilled water (making 38.5

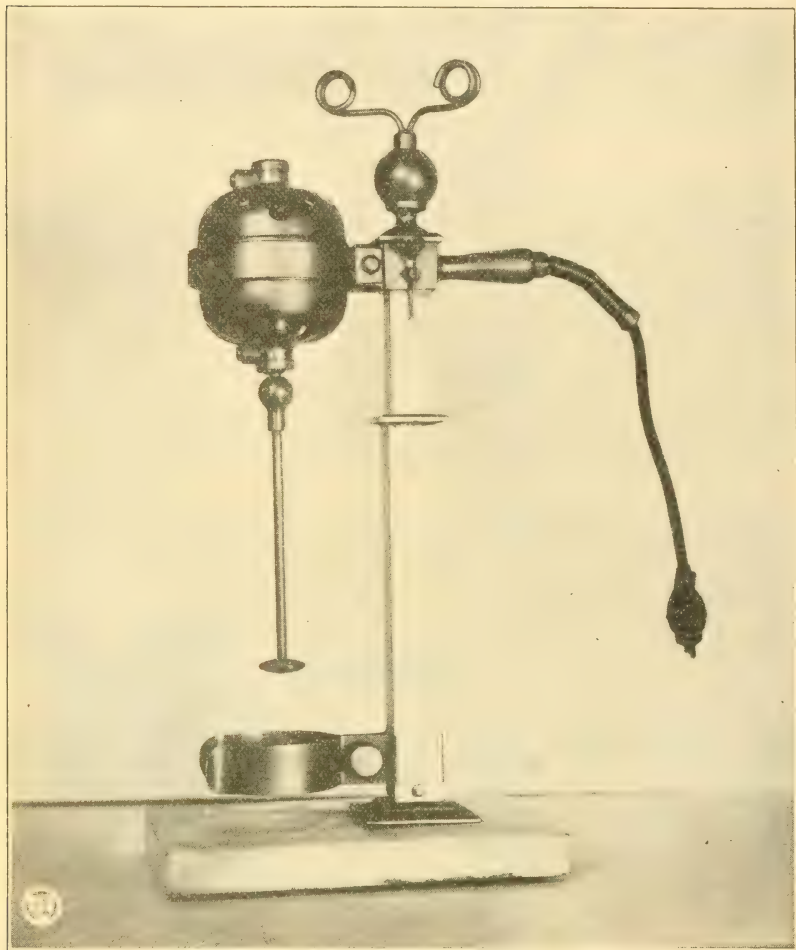


FIG. 13.—Electric agitator for mixing yeast solution.

cubic centimeters of yeast and water solution per loaf) and allowed to digest in the proofing cabinet at  $32^{\circ}$  C. ( $89.6^{\circ}$  F.) for half an hour, after which it is added to the salt-water solution in the crock and the two are thoroughly mixed.

Next, two-thirds of the flour is added to the salt, yeast, and water mixture in the crock and stirred, this amount being sufficient to make a fairly slack sponge. This sponge is placed in the proofing cabinet, where it is allowed to ferment for  $1\frac{1}{2}$  hours, after which



it is taken out and kneaded for approximately 3 minutes in the dough mixer. During this kneading, the sugar, the remainder of the flour, and enough distilled water are added to make a dough of the proper consistency.

The type of kneader in use at the experimental laboratory permits the accurate determination of the water absorption during the kneading process. With certain other types of experimental dough mixers on the market, it is not possible to determine accurately the water absorption during the kneading process. In the use of these types, a separate test for its determination is necessary. When

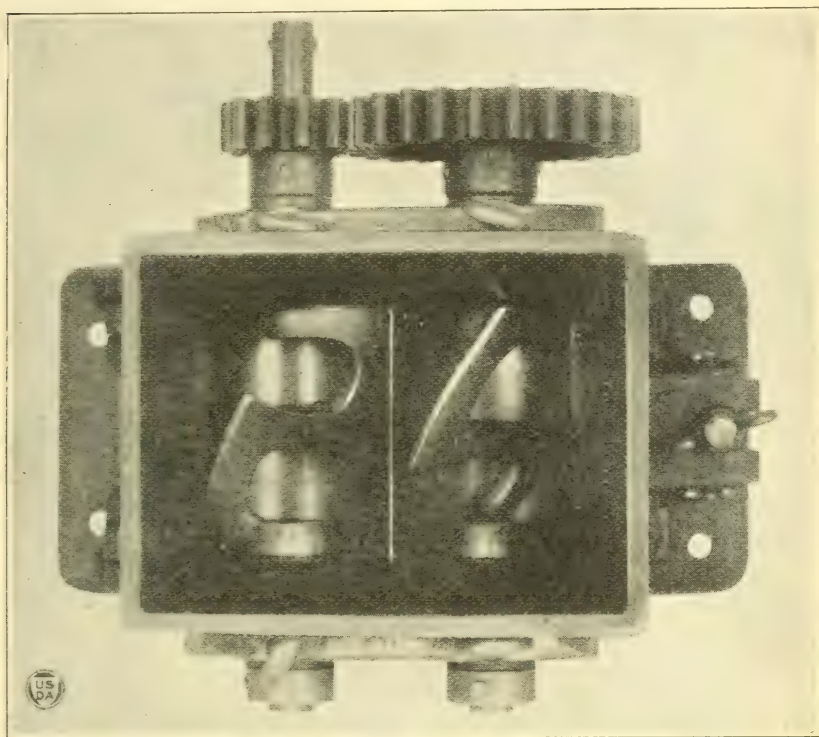


FIG. 14.—Dough-mixing machine.

16 or 18 tests are made each day, there is a considerable saving of time if the water absorption is determined during kneading, and it is believed that more accurate results are secured than when determined separately.

After kneading, the dough is placed in the proofing cabinet for 40 minutes, then it is "knocked down," and returned to the cabinet. After 25 minutes the dough is taken out, carefully moulded into the shape of a loaf, panned, pricked seven or eight times, and returned to the proofing cabinet, where it is allowed to remain until it rises to nearly its maximum volume, then put in the oven for baking. The time required for the loaf to rise varies from 50 to 100 minutes, depending on the activity of the yeast and the individual strength of the flour.

The temperature of the proofing cabinet during the fermentation period is kept at 32° C. This is accomplished by means of an automatic electric temperature-control device within the cabinet. The relative humidity of the air within the cabinet is kept at a high point (at least 85 per cent) by means of shallow pans of water placed in the bottom of the cabinet.

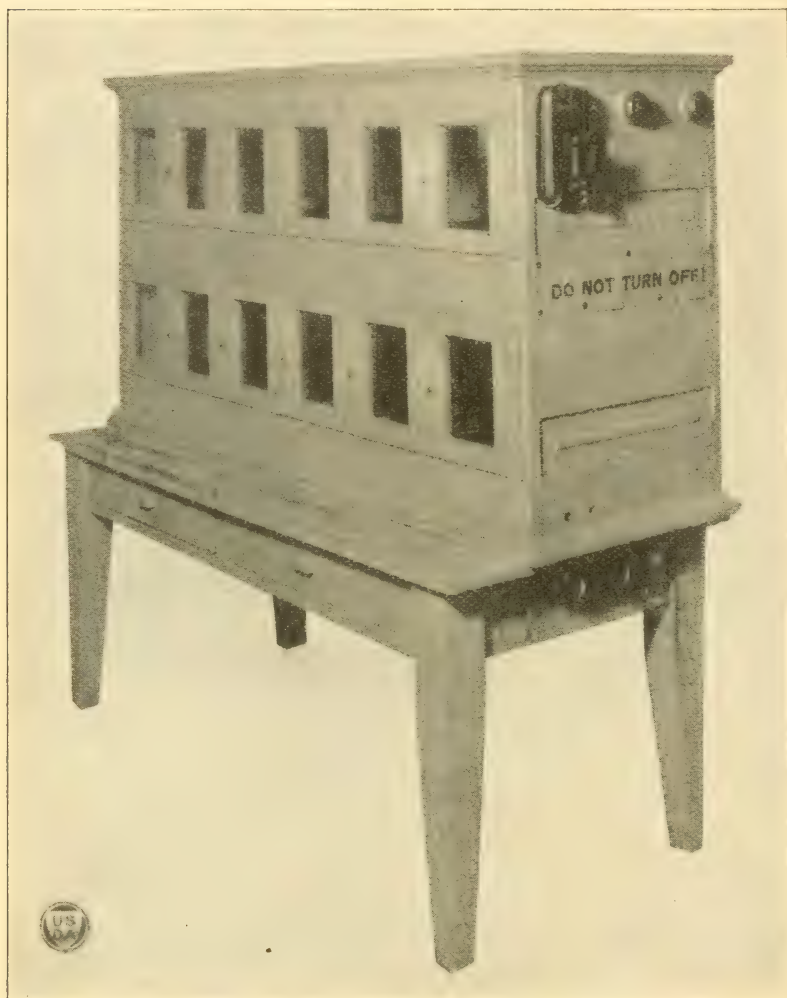


FIG. 15.—Dough-proofing cabinet.

The loaves are baked at a temperature of approximately 210° C. (410° F.) for 30 minutes. After cooling to nearly room temperature, the cubical content of the baked loaf is determined by use of the loaf-measuring device shown in Figure 17. The loaf is then weighed and placed in a storage cabinet until a convenient time for judging its color and texture, usually either the first or second day after baking. In judging, the bread is cut in half longitudinally and



compared with a standard or check loaf of known value. Figure 18 shows three different views of a loaf made by this method.

Record sheets (Fig. 19) are kept of each day's baking. On these are recorded for each sample tested, the time of sponging, kneading, panning, placing in oven, the percentage of water absorption, the volume of loaf in cubic centimeters, the color and texture scores, the shade of crumb, and the notation of any unnatural odors present or of anything else out of the ordinary observed during the handling or baking of the dough.

All baking tests are made in duplicate and the results recorded on the baking record sheet. The second baking is usually made on the day following the first baking for the purpose of minimizing error in judgment on the part of the baker in his treatment of the loaf and to serve as a check against the possibility of mistakes and

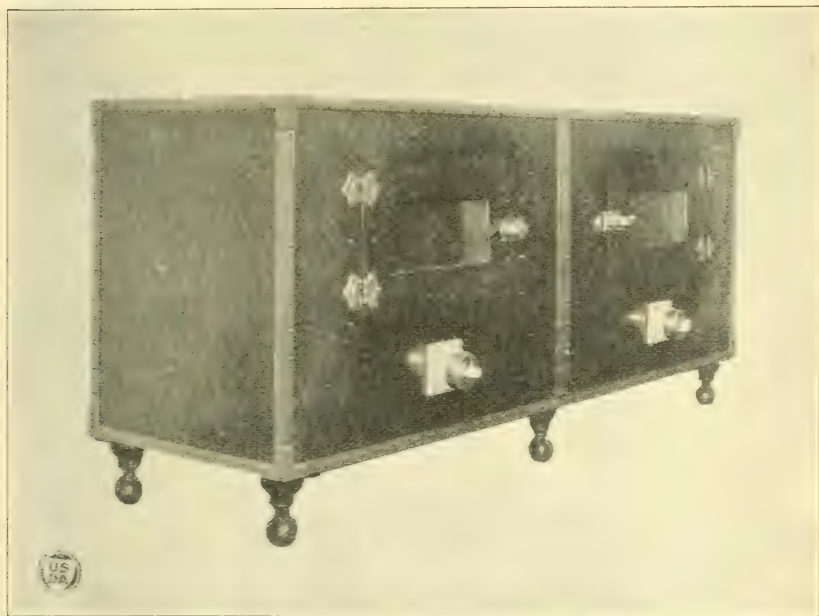


FIG. 16.—Electric baking oven.

any variation in the quality of the yeast. If the results from these two tests check closely, their average is taken as representative of the baking quality of the flour and is recorded on the laboratory card. If they do not check a third baking is made, provided sufficient material is available.

Each day a check loaf is baked from a selected lot of flour of known quality to indicate the presence of unusual conditions or changes in the quality of yeast which may cause a variation in the baking results and to serve as a standard for comparison in scoring for color and texture.

#### EXPLANATION OF THE VARIOUS BAKING OPERATIONS.

It is the object of the experimental baking laboratory to determine the relative differences in the quality of various samples of flour for bread-making purposes. To determine these differences accurately,

it is essential that the experiments be conducted under conditions that will produce results capable of comparison. This requires that the method and procedure of baking be standardized in every possible detail. The ingredients other than flour, namely the sugar, salt, and yeast, should be uniform both in quality and quantity in every test. The temperature and humidity of the proofing cabinet should

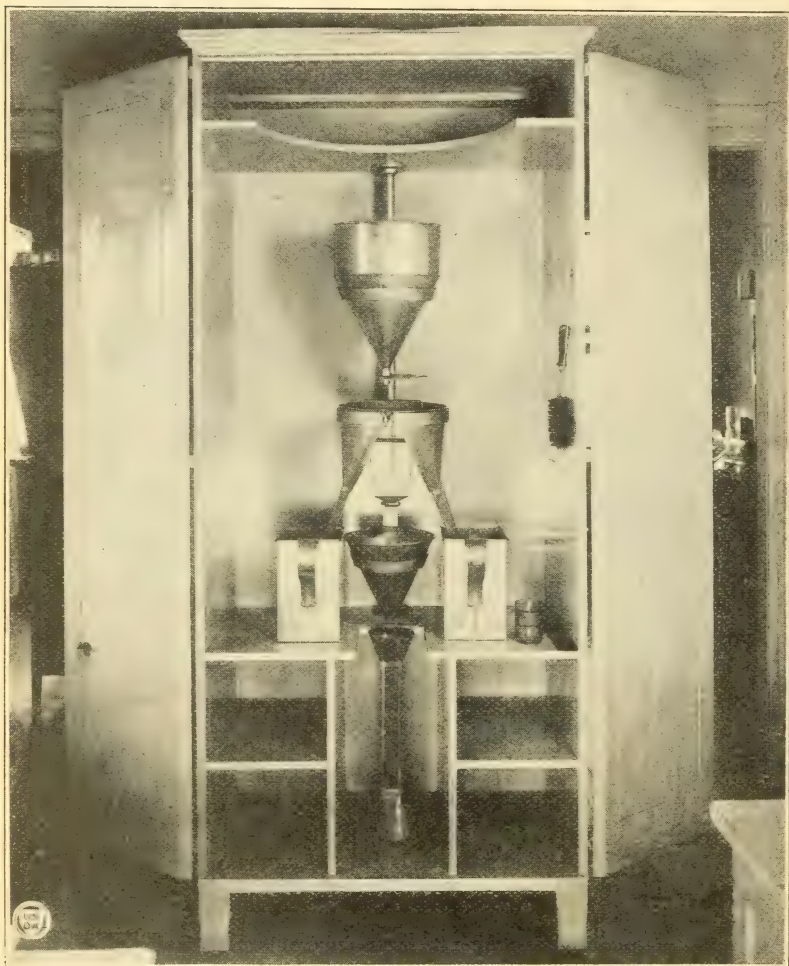


FIG. 17.—Loaf measuring device.

also be constant in order that any differences in baking results can not be attributed to the varying of these conditions. However, certain things, namely, the amount of water added to the dough and the length of proof, do not lend themselves to exact standardization because they are quality-denoting factors, and vary considerably for different samples, depending upon their inherent physical and chemical properties.



### KNEADING THE DOUGH.

Kneading the dough is a very important step in baking. During this operation the sugar and the remainder of the flour and water are added. Each batch of dough is given the same amount of kneading, that is, approximately 450 revolutions of the fast blade of the mixer. The number of revolutions is counted by means of a cyclometer attached to the driving shaft of the mixer. About 4 minutes elapse from the beginning of the kneading of one dough to that of the succeeding one.

It is during the kneading process that the baker gets his first idea of the baking strength of the flour. The manner in which the dough works up, its water-absorbing capacity, and its feel and appearance during the fermentation period all enable the baker to judge to some extent the quality of the gluten in the flour and the quality of bread that will be produced.

### KNOCKING DOWN, MOULDING, AND PRICKING.

Knocking down consists in folding the dough a certain number of times to make it into a compact mass, to liberate most of the gas,

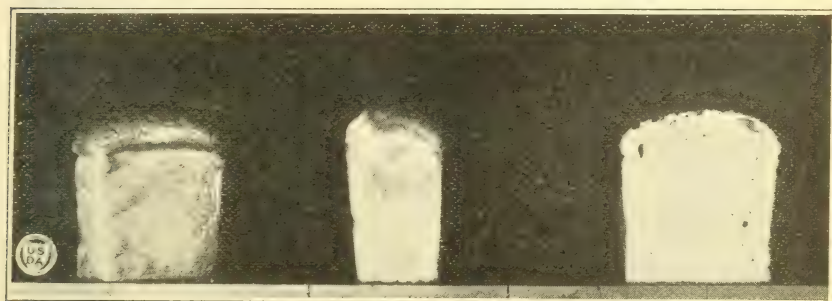


FIG. 18.—Views of experimental loaf.

and to bring other particles of dough which may not have already been acted upon in closer contact with the active yeast cells.

Moulding is the folding and shaping of the dough mass into the semblance of a loaf preparatory to panning, and is similar to knocking down, except that special care is taken to secure a smooth, unbroken top surface.

After being panned the dough is pricked with a stiff wire about the size of a hat pin to break the gas bubbles which may have formed near the top surface.

### WHEN TO PLACE IN OVEN.

The dough is placed in the oven when it has nearly reached its maximum expansion. This condition is judged from the surface appearance and shape of the dough and is reached about 50 to 100 minutes after the time of panning. The actual time required depends largely upon the quality of the flour and the activity of the yeast. About 50 minutes after the dough is panned, a close watch is begun for indications that the dough is in proper condition for baking. Doughs will expand two to three and one-half times their volume at panning time before they are ready for the oven.

The greatest expansion possible in dough comes when the elastic limit of the gluten is reached. If the dough is allowed to ferment to or beyond that limit before it is placed in the oven, the generation of more gas and its expansion due to the heat of the oven will cause a breaking down of the walls of the cells or pockets before they become permanently fixed by baking. This is especially true in the case of weak flours, and it results in a smaller, more poorly shaped, and coarser textured loaf than when the dough is placed in the oven before maximum expansion is reached.

In making these baking tests the aim is to place the dough in the oven just long enough before maximum expansion is reached so that the reserve strength in the gluten can take care of the expansion of gas in the dough due to the heat of the oven before the cell walls

No. ....		BAKING REPORT										DATE <i>Dec. 15 1922</i>	
SAMPLE	NUMBER	MOISTURE	WEIGHT	PAID	PUT IN OVEN	WATER USED	VOLUME OF	WEIGHT OF	COLOR	TEXTURE	WEIGHT OF	REMARKS	
<i>8546-20mm</i>	<i>1</i>	<i>10.20</i>	<i>11.20</i>	<i>12.25</i>	<i>1.50</i>	<i>1.98</i>	<i>21.00</i>	<i>4.95</i>	<i>41.5</i>	<i>89.0</i>	<i>crumbly</i>		
<i>8550-20mm</i>	<i>2</i>	<i>10.24</i>	<i>11.34</i>	<i>12.34</i>	<i>2.00</i>	<i>2.10</i>	<i>23.20</i>	<i>5.03</i>	<i>43.0</i>	<i>92.5</i>	<i>crumbly</i>		
<i>8551-17.5</i>	<i>3</i>	<i>10.28</i>	<i>11.38</i>	<i>12.43</i>	<i>1.54</i>	<i>2.07</i>	<i>22.30</i>	<i>5.09</i>	<i>42.5</i>	<i>87.0</i>	<i>crumbly</i>		
	4												
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	24												
REMARKS		<i>#2 slight musty odor upon removal from oven</i>											

FIG. 19.—Baking record sheet. (Size, 8½ by 11 inches.)

become fixed by baking. In commercial baking the dough is usually placed in the oven some time before it has expanded to its maximum volume, in order that a finer textured bread may be produced than would result if it were allowed to expand further.

#### WATER ABSORPTION.

Considerable experience is required in adding the correct amount of water to a dough. If too much water is added, the dough loses stability and produces bread of a coarser texture: If too little water is added, a smaller and more solid and closer-textured loaf is produced.

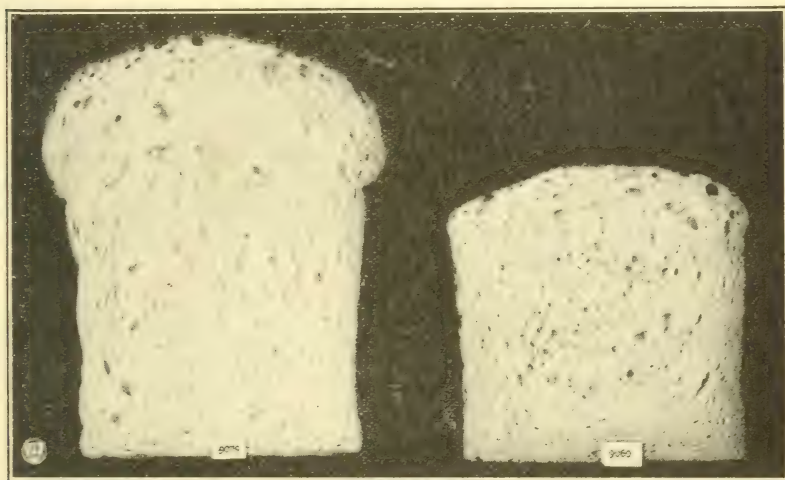
The absorption or quantity of water used in making the dough is of importance from the commercial standpoint since, other things being equal, the more water that can be worked into a given weight of flour, the greater the weight of bread it will produce. Some of



the water contained in the dough is lost during baking, but there is a fairly well-defined relationship between the weight of the resultant loaf and the water absorption of the flour.

For ordinary flours, the total quantity of water added previous to kneading is 180 cubic centimeters, which includes the 150 cubic centimeters in the salt solution and the 30 cubic centimeters in the yeast mixture. The addition of more water is usually necessary during kneading to give the dough a proper consistency. Occasionally, a flour is found in which the 180 cubic centimeters of water in the salt and yeast solution is more than enough to give the dough its proper consistency. When this occurs, care is taken that a smaller quantity of water is used in making up the sponge in the duplicate test.

In calculating the percentage of water absorption, no account is taken of the natural moisture content of the flour. The percentage



Loaf volume 2,620 cubic centimeters.

Loaf volume 1,670 cubic centimeters

FIG. 20.—Differences in volume of loaf made from the same quantity of flour.

of water absorption represents the weight relationship of the water absorbed to the 340 grams of flour used in the test.

### SIGNIFICANCE OF FACTORS DENOTING QUALITY.

#### LOAF VOLUME.

When the dough is allowed to ferment to the point where the loaf of greatest size possible is produced, the loaf volume, if uniform quantities of flour, yeast, salt, and sugar are used, may be considered an expression of the relative strength of the flour. It is not desirable to allow the fermentation of dough to proceed to the maximum point of expansion in commercial or household baking, but flour which will yield large loaves under this test will make bread of good quality and texture when baked in the usual way, and it will stand more neglect during the process of bread-making as well. Large loaf volume when associated with good texture is a desirable quality.

Figure 20 shows two loaves of bread made from the same quantity of flour showing a difference of over 1,000 cubic centimeters in loaf

volume and illustrates the wide variation in volume which may occur between a weak and a strong flour.

The volume of the loaf is determined after it has cooled by use of the volume-measuring device shown in Figure 17. The large compartment of this device is of a known cubical content and is greater than the maximum cubical content of any loaf to be measured. The loaf is placed in the compartment, after which flaxseed is allowed to run through a funnel into the compartment until it is heaping full. The flaxseed above the rim of the compartment is stroked off in the same manner as the excess grain from a test-weight kettle in making a weight per bushel test. The flaxseed is then drawn out through a valve at the bottom of the box into a funnel supported above a flask graduated in units of 10 cubic centimeters. The difference between the volume of the flaxseed in the flask and the known volume of the flask represents the volume of the loaf, which is read directly on the neck of the calibrated flask.

Some laboratories use mustard, rape, or other small seed instead of flaxseed in measuring loaf volume, and others calculate volume merely from measurements of the loaf.

#### WEIGHT OF LOAF.

The loaves are weighed after they have cooled to nearly room temperature and the weights are expressed in grams. If the baking is done under uniform conditions the weight of the loaf is of value in calculating the number of unit loaves which can be produced from a given quantity of flour.

#### COLOR OF CRUMB.

Color and texture scoring is usually done the day immediately following the completion of the duplicate baking, because at that time the loaves have sufficiently cooled to withstand cutting in half longitudinally without injuring the texture. After the loaves are cut, they are lined up and their respective color values determined by comparison with the standard loaf, the color value of which is determined arbitrarily. The general appearance of the loaf and the color shade of the crumb, such as creamy, slightly creamy, creamy-gray, and gray, are also noted and recorded.

The flour selected for making the standard loaf should score very near the average in color, as this facilitates a more accurate comparison. In order that more consistent color comparisons with the standard loaf may be made from day to day, a clear white color is taken as the ideal, regardless of the class of wheat from which the flour was milled. A very creamy or dull gray colored crumb receives a lower color score than the lighter shades of these colors or their combinations.

Because of the variable intensity of the sunlight, it is rather difficult to secure consistent results in color scoring from one day to another, and for this reason some laboratories prefer to use artificial light in color scoring. In this laboratory natural light is used in color scoring, which is always done in the same room, with the loaves placed in similar positions, and viewed from the same angle to minimize the effect of varying light conditions which may occur from day to day. It is preferable for the person scoring to use two standard loaves of different shades of crumb, one of a creamy shade and the other of a creamy-gray shade.

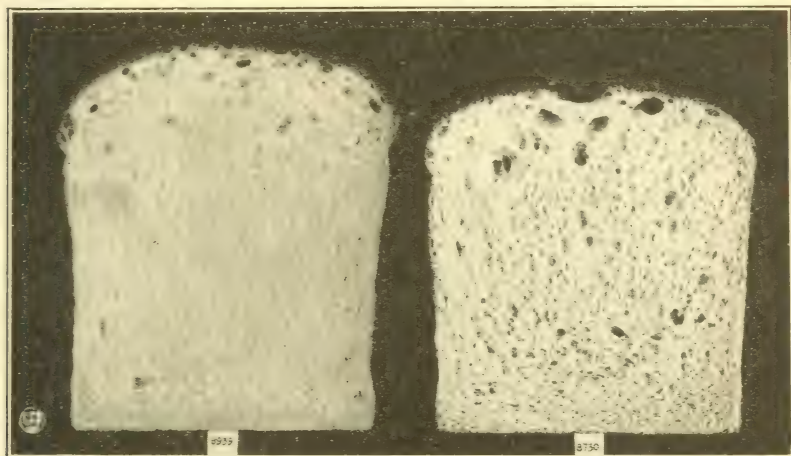


In scoring for color and texture, 100 represents the maximum or perfect score, 96 to 100 excellent, 91 to 95 good, 86 to 90 fair, 81 to 85 poor, and 80 or lower very poor. The different shades of color found in bread are white, gray, and creamy in varying degrees and combinations. Other systems of scoring bread are in use in which color and texture are judged in a somewhat different manner than described here. One such system particularly worthy of mention is that recently devised by the American Institute of Baking and adopted as the official system for use by the baking industry.

Because of the variability of human judgment and of natural light conditions, the method used at present for arriving at the numerical color and texture scores of the bread is not entirely satisfactory, but no mechanical device is known which will give satisfactory results.

#### TEXTURE OF CRUMB.

The method used in scoring for texture is similar to that for color. An arbitrary texture value is given to the standard loaf and the



Texture of crumb score, 93.

Texture of crumb score, 85.

FIG. 21.—Differences in texture of crumb.

other loaves are given comparative scorings, as previously described for color. The standard loaves may vary slightly from day to day, because of slight differences in manipulation, strength of yeast, and temperature of the proofing cabinet, but this variation does not affect the value of the standard loaf for use in scoring for texture.

The texture score is dependent upon several related factors, of which uniformity, number and evenness of distribution of the cavities or cells, and thickness and character of cell walls are the more important. For a loaf of good texture the cells should be small, uniform in size, and thin-walled. The walls should be tender but not easily crumbled, and the bread should be soft to the touch and possess resiliency. Figure 21 shows two loaves of about the same loaf volume, the one having a good and the other a poor texture. Texture and loaf volume are closely associated in that the one may be improved at the expense of the other.

## BAKING METHODS USED IN OTHER LABORATORIES.

Some experimenters use the straight-dough method of baking bread. The procedure in this method is substantially as follows:

Formula for each loaf:

	Grams.
Flour.....	450
Sugar.....	16
Salt.....	7
Yeast (compressed).....	10
Water (distilled), based on percentage of absorption.	

The salt and sugar are dissolved in part of the water and placed in the mixer with the flour. After starting the mixer the yeast is added with the remainder of the water and mixed for two or three minutes, the amount of mixing depending upon the smoothness of the dough. The dough is then removed from the mixer and placed in the fermentation cabinet at 28 to 30° C. with sufficient humidity to prevent crusting.

The length of time before the first punch, varies with the type of flour used, but is seldom longer than 100 minutes, the second punch in 40 to 45 minutes, and the third in 15 minutes, at which time the dough is panned and allowed to proof at 36 to 38° C. until ready for the oven.

At the time of panning, 170 grams of dough are cut from each batch and placed in expansion jars which are put in the proofing cabinet. The dough in the jar is allowed to ferment until its maximum expansion is reached, readings being taken every 15 minutes until it falls.

Each loaf is baked from 30 to 35 minutes at 220° C.

Some laboratories do not run expansion tests, so from 100 to 110 grams less of flour are used and the other ingredients except possibly yeast, are reduced in proportion.

The points or quality factors for which the baked loaf is judged and the method of judging are substantially the same as those described under the sponge method.

The methods of baking in use at some of the State experiment stations are a modification of either the straight dough or sponge method. At the University Farm, Minnesota Experiment Station, an apparatus known as the expansimeter is used for determining the expansibility of the dough.<sup>4</sup> A similar apparatus is used by the Kansas State Experiment Station.<sup>5</sup> In commercial bakeries and in home baking the processes of bread making vary considerably in certain details and in equipment.<sup>6</sup>

A few experimental baking laboratories endeavor to approximate the commercial formulas and type of loaf produced. The bread produced in the laboratory of the Bureau of Agricultural Economics is coarser in texture than the commercial loaf, because the dough is allowed to ferment almost to the point of maximum expansion before it is put into the oven. This is done because the differences in quality existing between samples can be determined more accurately when the maximum expansion capacity of the dough is approached. The

<sup>4</sup> Described in Journal of Industrial and Engineering Chemistry, vol. 8, No. 1, p. 53; A Method for the Determination of the Strength and Baking Qualities of Wheat Flour, by C. H. Bailey. January, 1916.

<sup>5</sup> Described in Kansas State Agricultural College Experiment Station Bulletin 177; Milling Tests of Wheat and Baking Tests of Flour, by J. T. Willard and C. O. Swanson. 1911.

<sup>6</sup> Full instructions for home baking are given in United States Department of Agriculture, Farmers' Bulletin 1136; Baking in the Home, by Hannah L. Wessling. 1923.



flour that produces the best bread in the testing laboratory is most likely to produce the best quality of bread when baked with commercial formulas and equipment.

### CHEMICAL DETERMINATIONS.

In connection with the milling and baking experiments described above, certain chemical determinations are made. These determinations are essential for several reasons. In the first place, they show the investigator whether the differences in flour strength indicated by the baking test are true or are due to the treatment received by the wheat during milling or in the subsequent baking process. In milling the same sample of wheat into more than one grade of flour there will naturally be a difference in the composition of the flour as a result of the distribution of the flour yielding material in the wheat kernel. It is essential in making comparative studies that the composition be known so that the same grade of flour be used throughout. Comparative results from studies on patent flours, for instance, should by no means be included with data from studies on straight or other grades of flour.

Second, the results of chemical studies offer constructive suggestions to the miller in the matter of handling wheat at the mill and to the baker in handling flour in the bakery. Third, inasmuch as no chemical test or group of tests has been developed which will give conclusive evidence as to the kind of bread which may be produced from any given lot of wheat or wheat flour, the correlation of the results of chemical and physical determinations with the results of baking and milling tests throws light upon some of the causes for differences in the strength of wheats, and may eventually lead to the development of simple chemical tests for determining flour strength.

There is no unanimity of opinion in answer to the question as to what chemical constituents are of principal importance in regulating the baking strength of flour. Current among the factors associated with quality of flour are: The proteins of the flour, the nature and quantity present; the influence of acids, bases, and salts upon the physical properties of the proteins; the acidity of the flour; the nature and amount of the mineral salts already present; and the kind of food supplied the yeast plant during the fermentation of the dough, as well as the nature of the fermentation products (sugars and proteins) developed by diastatic or proteolytic enzymes.

Wheat flour contains five distinct proteins: An albumin, a globulin, a prolamine (gliadin), a glutelin (glutenin), and a proteose. In addition to these compounds, amino acids are also found in the wheat and to a lesser extent in the flour. Albumin, globulin, and amino acids are found chiefly in the embryo. Their presence in flour is due to the impossibility of entirely separating the germ from the endosperm during the process of roller milling. The gliadin and glutenin constitute from 85 to 88 per cent of the total protein of high-grade flour. The proportion of gluten to crude protein in whole wheat is somewhat lower than this. When wheat flour is made into dough these two compounds, commonly called gluten, absorb approximately two to three times their combined weight of water, forming a more or less coherent or tenacious mass. It is the expansive force of the gas generated by the yeasts during the fermentation of the



dough which becomes entrapped in the particles of gluten that causes the dough to rise in the process preliminary to baking. Like all other proteins, gluten becomes dry and hard upon exposure to heat, so that the shape of the loaf becomes set soon after it is placed in the oven.

It is plain that wheats with a high, medium, or low protein content will, in milling, yield a flour having a correspondingly high, medium, or low protein or gluten content. Except those wheats which have an excessively high protein content, and which as a rule yield flours of less strength than those having medium protein content, the average of a large number of samples within the same class of wheat shows that the baking strength of a high-grade flour is usually high if the percentage of protein is high, and low if the percentage is low. Greater variations are found in wheats with a low or high percentage of protein than in those with a medium percentage. These variations, moreover, fully illustrate the fact that the results of total crude protein determinations are not always indicative of baking strength. This lack of relationship is due apparently to the physical properties of the gluten proteins, as the same kind of protein from different classes and grades of wheat is chemically identical.

The percentage of crude protein in both wheat and wheat products is obtained by multiplying the percentage of total nitrogen as determined by the Kjeldahl method (p. 36) by the factor 5.7. The term "crude protein" is used because wheat and wheat products all contain nitrogen-bearing substances which are not protein or glutinous in nature. By use of the factor 5.7 the approximate percentage of true gluten present in the sample is determined, as it has been found that true gluten contains approximately 17.6 per cent nitrogen. The apparatus for making crude protein tests are pictured in figures 22, 23, and 24.

The methods used to arrive at the percentage of crude gluten vary. The most common method (described in full on page 40) is to mill out the flour from the wheat and make a dough ball of the flour by adding water. The dough ball is then washed under a stream of water until everything but the gluten is gone. This remains as a tough, elastic mass known as wet gluten. When dried in an oven at 100° C. or 212° F. for 24 hours, or until it ceases to lose weight, it is called dry gluten. When carried out by the same person under the same conditions this method is fairly accurate as a means of determining the relative percentage of crude gluten present in the sample of flour under examination. An indication of the quality of the crude gluten may also be had by observing the color, consistency, and the general condition of the washed gluten.

It does not show, however, the total amount of gluten in the wheat itself. The amount of flour extracted from any lot of wheat varies according to the practice of the miller. Because of the distribution of nitrogen compounds within the kernel, the amount of gluten in any given sample of flour varies directly with the percentage extraction, being higher as a rule in the straight flours. It will be seen, therefore, that results obtained by making gluten determinations on flour are of questionable value as an index to the quantity of the gluten of the original wheat kernel unless the determination is made on a straight 100 per cent flour. As was pointed out above, at the present time there is no absolute method for determining directly

the amount of true gluten in flour, the practice of determining the total amount of nitrogen and multiplying by the factor 5.7 offers the closest approximation to the true gluten content.

Although other factors contribute toward quality in flour, the making of a large, light loaf of bread depends primarily upon an adequate quantity and quality of gluten in the flour. In general it may be said that the most desirable quality of gluten is that of



FIG. 22.—Standard solutions. Automatic burettes in foreground. Electric stirring apparatus for general color determinations and acidity of corn.

decided coherence or tenacity when subjected to a reasonable stretching force. What the conditions are which function in the making of a strong flour is a question of the greatest importance, and up to the present time it has remained unsolved, although decided differences in gluten quality from wheats from different sources have long been known.

It is sometimes of value to estimate the proportion in which the individual proteins are present either in wheat or flour. In the experimental laboratory the amount of gliadin is determined by

boiling the wheat or flour in 50 per cent alcohol, by volume, for 3 hours.<sup>7</sup> Bailey and Blish's method for determining the salt soluble proteins, i. e., albumin and globulin, is also used. Glutenin is determined by deducting the sum of the potassium sulphate soluble

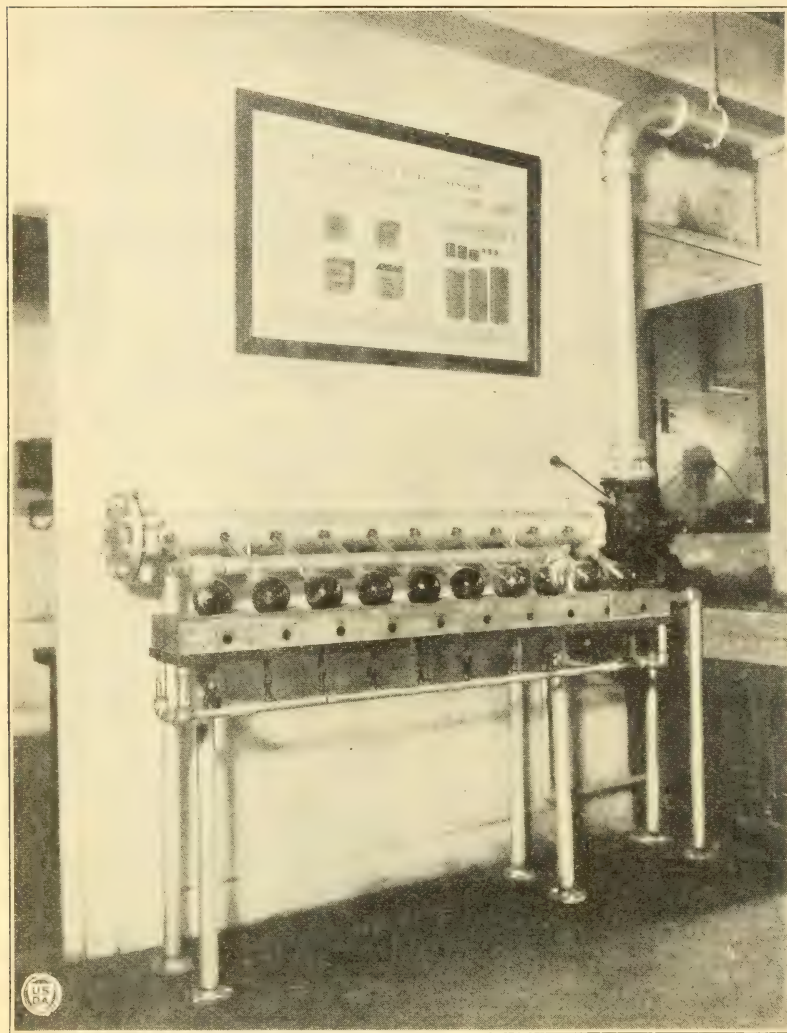


FIG. 23.—Kjeldahl digestion apparatus for the determination of protein in cereal and cereal products. Digestion flasks, with material, in position.

nitrogen and the alcohol soluble nitrogen from the total nitrogen and multiplying the difference by 5.7.

The acidity or reaction of the flour or wheat is a very important factor, as it controls, to a large extent, the quality of the flour. Through the proper H-ion concentration the elasticity of the gluten

<sup>7</sup> See Journal of Biological Chemistry, vol. 23, No. 1, page 345; Concerning the Identity of the Proteins Extracted from Wheat Flour by the Usual Solvents by C. H. Bailey and M. J. Blish, 1915.



is increased as well as the activities of the yeast. The optimum reaction corresponds to a  $P_H$  of approximately  $-5.0$ . For choice flours this figure is a little higher, and for the poorer grades a little lower.

The percentage acidity of flour ordinarily diminishes with increasing refinement and grade. Exceptions to this rule are found,

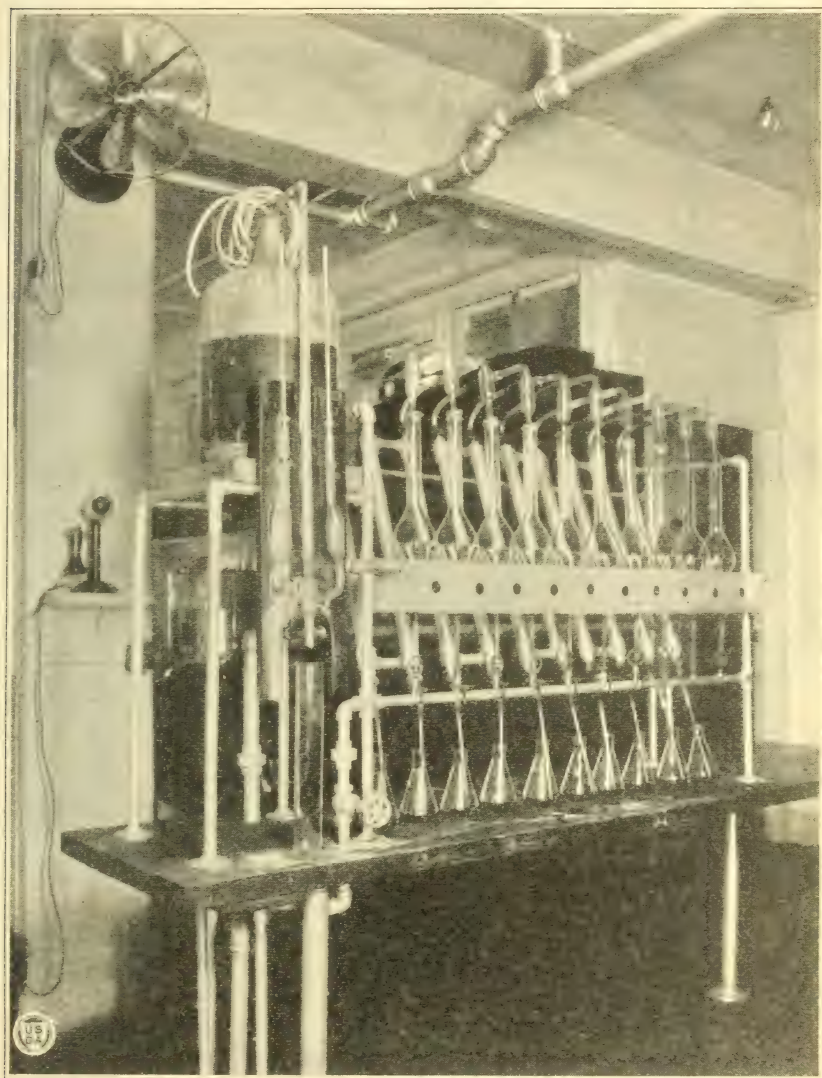


FIG. 24.—Kjeldahl distilling apparatus with glassware in position for making test. Supply of caustic soda and automatic pipette to left of the still.

however, in flour made from unsound wheat, or in flour milled from wheat containing noticeable quantities of inseparable foreign material. It is necessary that a normal acid limit for each grade of flour in sound condition be established before its acidity value can be used to judge its condition. As a rule, sound flours are low in acidity, whereas flours which have spoiled or are made from spoiled wheat

usually show a high percentage of acidity. It is understood of course that a low-grade flour, even when sound, will have a relatively high acidity, due to its inherent nature.

The ash determination is more generally employed for detecting the relative grade of wheat flour. Usually the higher the grade of flour from a given wheat, the lower is the percentage of ash, indicating that a less amount of the outer portion of the wheat kernel is in the flour. It should be borne in mind, however, that a low percentage of ash is not an absolute indicator of high quality, as the soft wheats, as a class, which are relatively weaker on the average than the hard wheats, have low ash content. It should also be pointed out that flours milled from durum wheats run high in ash content, and that this does not preclude their making a fair loaf of bread. Considering individual samples, the total ash content appears to be in no way a distinguishing characteristic of flour made from different varieties of wheats or from wheats of different classes.

The ash content of flour is always less than that of the wheat from which it is milled. In the product of the commercial mill there is less ash in the patent than in the lower grades. In milling for straight flour the ash content depends to some extent upon the closeness of grinding.

The gasoline color test of flour (p. 43) is of especial interest when made upon flours milled from durum wheats. This is doubly true when the test is interpreted in conjunction with the total protein and ash content of the flours. Durum wheats, with their relatively high protein content, generally speaking, lend themselves in a large measure to the manufacture of macaroni. The usual baking test with flour from this class of wheat gives good evidence as to the quality of gluten in the flour, and an ash determination indicates the grade of flour. The color of durum flours, cream to creamy yellow, is decidedly below the usual standard for bread-making purposes. On the other hand, from a macaroni standpoint the high color, so detrimental from a breadmaking standpoint, is just what is desired, and it is quickly and decisively brought to light by making a gasoline color test.

Correct moisture determinations are of great importance, inasmuch as under the pure food and drugs act no flour can carry more than 13.5 per cent moisture. Water in excess of this figure is considered an adulteration. In making moisture determinations on whole grains for the purpose of grading or in connection with the tempering of wheat for milling, the Brown-Duvel moisture tester is used.<sup>8</sup> In making moisture determinations on ground material or flour the electric oven is used for a period of four to five hours at a temperature of 108° C.

#### DESCRIPTION OF METHODS OF CHEMICAL DETERMINATIONS.

The methods for making chemical determinations hereafter described are substantially those of the Association of Official Agricultural Chemists. Where modifications of these methods are described, it is believed that they do not vitiate the accuracy of the determination. They have been used by reason of their greater simplicity, ease of manipulation or lessening the time element in

<sup>8</sup> United States Department of Agriculture Handbook of Official Grain Standards, p. 34, 1922.



conducting the analysis. In all flour analyses involving questions of food law enforcement, the methods of analysis as adopted by the Association of Official Agricultural Chemists should be used.

The order in which the methods of making the various tests are described below does not necessarily follow the order in which they were just discussed. In fact, on account of the limited use made of some methods no discussion of them was presented at all.

On the contrary, it has been attempted to picture the logical sequence of tests a sample would receive as it passes through the laboratory. This of course does not mean to imply that each and every test is applied to all samples. In many cases only a few tests are made on a sample, for example, moisture, ash, and crude protein tests. The methods follow in detail.

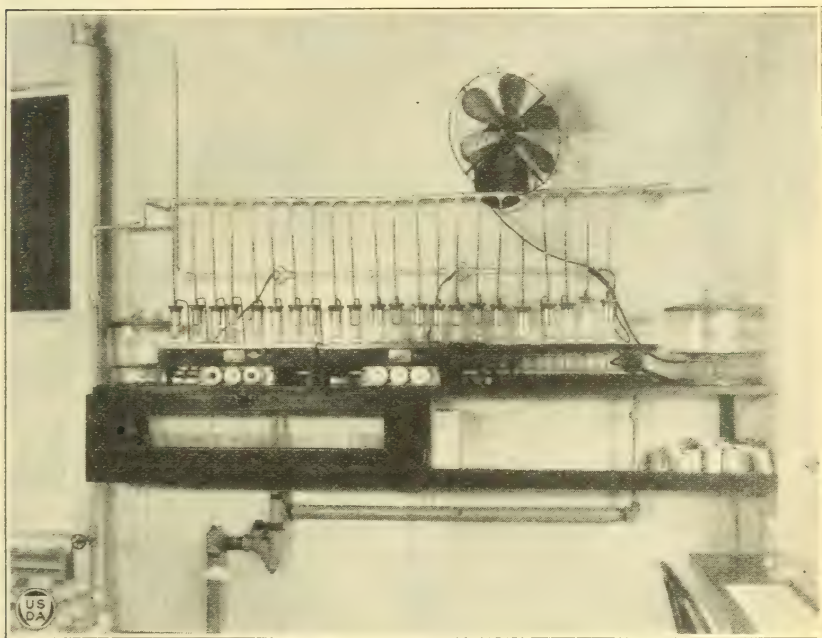


FIG. 25.—Fat extraction apparatus.

#### MOISTURE IN FLOUR AND WATER.

In making a moisture test of flour, duplicate 5-gram portions of the sample are weighed into tared aluminum moisture boxes which are supplied with closely fitting covers. The sample is then heated in a triple walled relay-controlled electric oven at a temperature of  $108^{\circ}\text{C}$ . for a period of approximately five hours. The bulb of the thermometer should be on a level with the sample. At the end of the heating period the covers are placed on the moisture boxes and they are removed to a desiccator to cool for approximately 45 minutes. They are then rapidly weighed and the loss in weight calculated as moisture.

The boxes used are seven-eighths of an inch high and are  $2\frac{1}{2}$  inches in diameter. Ovens for making moisture determinations are pictured in Figure 26.



The moisture determination on whole wheat is made according to the recommendations in the Handbook of Official Grain Standards.

*Preparation.*—Standardize the heating time for making the moisture test as follows: Place 450 cubic centimeters of oil (the same as is used in the moisture test) into the flasks of each compartment of the tester. Insert the thermometer and regulate the mercury bulb so that the top is just flush with the surface of the oil. Connect the flasks with the condensing tube, replace the covers, light the gas, and note the time. Apply heat until the temperature of the oil reaches  $175^{\circ}\text{C}$ . and again note time. The time elapsed should be about 20 minutes. If more than 20 minutes is required to reach a temperature of  $175^{\circ}\text{C}$ ., the holes in the bases of the burners should

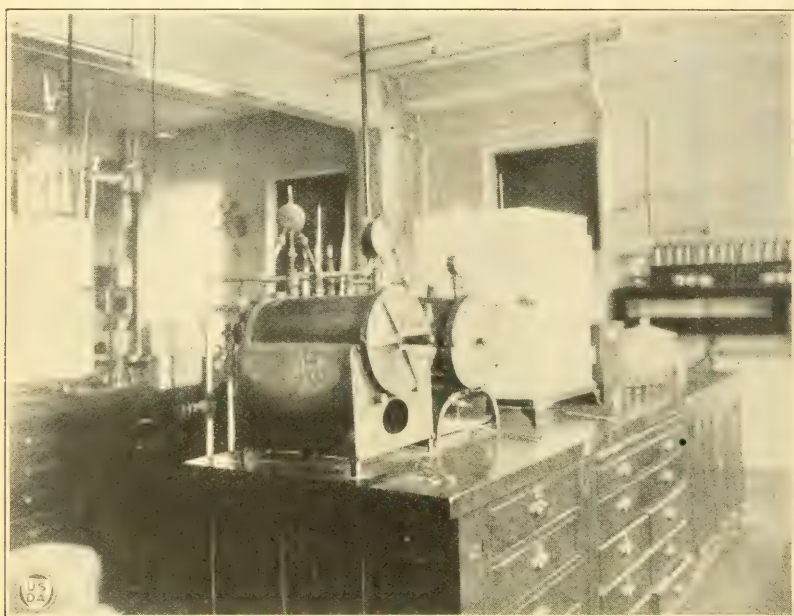


FIG. 26.—Ovens for making moisture tests. Vacuum ovens in foreground. Triple-walled electric oven in background.

be opened; if less than 20 minutes the holes should be closed. The holes may be closed by lightly tapping the top of the base with a small hammer or any blunt instrument. They may be opened with a suitable reaming tool, such as a fine brooch, used by jewelers. Trial tests should be continued until the required time for heating all compartments does not vary more than one-half minute from 20 minutes; that is, not less than  $19\frac{1}{2}$  minutes nor more than  $20\frac{1}{2}$  minutes, should be necessary to raise the temperature of the 450 cubic centimeters of oil to  $175^{\circ}\text{C}$ . If a gas-pressure regulator is used, a uniform heating time is assured. If, however, this instrument is not available, it will be necessary for the operator to fix in his mind the flame necessary to heat the tester in the proper time, and, in case the gas pressure varies, to compensate for this by adjusting the keys and air valves at the base of the burner.

*Determination.*—Place 100 grams of wheat, together with 150 cubic centimeters of oil, in the distillation flask and shake with a slight whirling motion until the two become well mixed. Insert the thermometer so that the mercury bulb will be immersed approximately three-fourths of its length. Place the flask in the heating compartment, connect with the condenser tubes, have a stream of water running through condenser tank, place graduated tubes under the condenser tubes, and start flame. Extinguish the flame when the thermometer reaches  $180^{\circ}$  C. for wheat, and let the mercury recede to  $160^{\circ}$  C. before making reading. It is absolutely necessary to adhere to these directions to secure correct results.

In this connection it should be stated that up to this time the gas-heated Brown-Duvel machine is the only type that has given



FIG. 27.—Bench showing Boerner sampler, test weight per bushel apparatus, and balance for weighing samples.

satisfactory service for moisture tests. The electrically heated machines have not been perfected to the point where the heat units (wattage) can be conveniently controlled to give accurate results. The Brown-Duvel moisture tester with gas-pressure regulator in place is shown in Figure 29.

#### ASH IN FLOUR AND MEAL.

To make an ash determination of flour or meal, weigh duplicate 3-gram portions of the material into a previously ignited and weighed porcelain combustion capsule. Place in an electric muffle and heat gradually until the color of the inside of the oven is a dull red. Maintain at this temperature overnight. The ash is not fused by this method and is white to gray-white in color. In the morning remove to a desiccator and allow to cool. Reweigh the capsule and consider the gain in weight as ash.

The combustion capsules are the same type as are used in coal analysis, have a capacity of 25 milliliters, are 20 millimeters high, and 40 millimeters in diameter. Ovens and crucibles for making ash determinations are pictured in Figure 30.

#### ACIDITY IN FLOUR.<sup>9</sup>

To determine the acidity of flour, weigh 18 grams into a 500 cubic centimeter Erlenmeyer flask and add 200 cubic centimeters of distilled water, previously freed from carbon dioxide by boiling. Place the loosely stoppered flask in a water bath kept at 40° C. for 10 minutes, shaking repeatedly. Remove the flask and with an occasional shaking allow it to stand at room temperature for one hour.

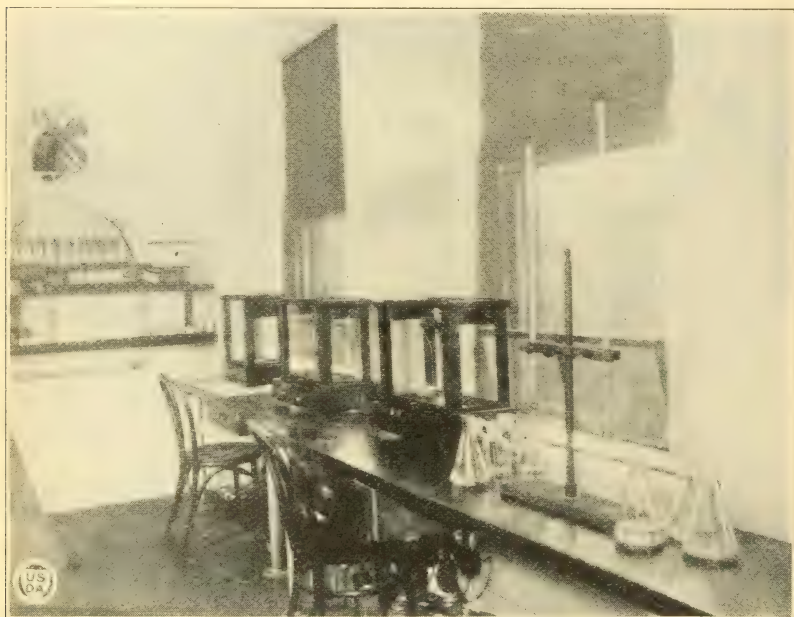


FIG. 28.—Bench showing analytical balances used for weighing small samples of grain, flour, etc., for the purpose of making ash, moisture, protein, and other tests.

Filter through a dry folded filter, rejecting the first 10 cubic centimeters and receiving the succeeding 100 cubic centimeters in a graduated flask. Titrate the filtrate with N/20 sodium hydroxid, using 2 cubic centimeters of carefully neutralized phenolphthalein in alcohol as an indicator. Each cubic centimeter of N/20 sodium hydroxid represents 0.005 gram of lactic acid or 0.05 per cent acidity as such.

#### PROTEIN IN WHEAT AND FLOUR.

*Reagents.*—N/10 hydrochloric acid; N/20 sodium hydroxide; concentrated sodium hydroxide (600 grams per liter) to which has

<sup>9</sup> Official and Tentative Methods of Analysis of the Association of the Official Agricultural Chemists, revised to November 1, 1919, p. 167.



been added sodium thiosulphate<sup>10</sup> in the ratio of 1 gram to every determination; concentrated sulphuric acid, free from nitrate, specific gravity 1.84; mossy zinc, mercuric oxide (yellow), 1 per cent alcoholic solution of methyl red indicator, C. P. sodium sulphate.

*Determination.*—Place 2 grams of the sample in a Kjeldahl flask and determine the nitrogen as follows: Add approximately 0.7 gram of mercuric oxide, or its equivalent in metallic mercury, 10 grams of sodium sulphate, and 25 cubic centimeters of sulphuric acid to the flask and place on the digestion frame in an inclined position. Adjust the heat so that the contents of the flask will be covered and digest for 15 or 20 minutes after the solution becomes colorless or straw-colored. Cool and dilute with 200 cubic centimeters of dis-

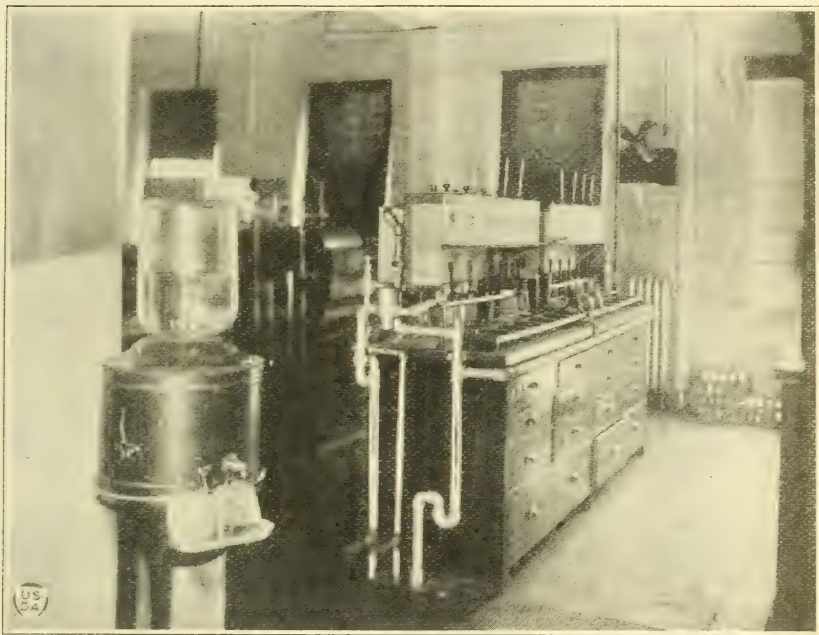


FIG. 29.—Moisture tester for rapidly determining the moisture content of cereal grains and other substances. Gas-pressure regulator on gas supply line in foreground.

tilled water. Add a small piece of mossy zinc to prevent bumping, then sufficient saturated sodium hydroxide solution containing 1 gram sodium thiosulphate per determination to make the reaction strongly alkaline, pouring this solution down the neck of the flask without mixing until after connecting with the distilling bulb. Distill off the ammonia into a definite amount (20 or 25 cubic centimeters) of standard N/5 hydrochloric acid which has been placed in the receiving bottle and diluted with about 50 cubic centimeters of neutral distilled water. Continue the distillation for 30 to 50 minutes when 100 to 150 cubic centimeters of distillate will have been collected. Titrate to the neutral point with standard N/10 sodium

<sup>10</sup> The use of sodium thiosulphate as a mercury precipitant is not as yet used by the Association of Official Agricultural Chemists, but has been found to give just as accurate results, and furthermore has the threefold advantage of being less expensive than potassium or sodium sulphide, is easier to get into solution, and when combined with the caustic soda speeds up the test.

hydroxide solution, using methyl red as the indicator. Correct for blank on reagents and calculate first, the percentage of nitrogen, and then the percentage of protein by multiplying the per cent of nitrogen by 5.7. The apparatus for making protein tests is shown in Figures 22, 23, 24, and 31.

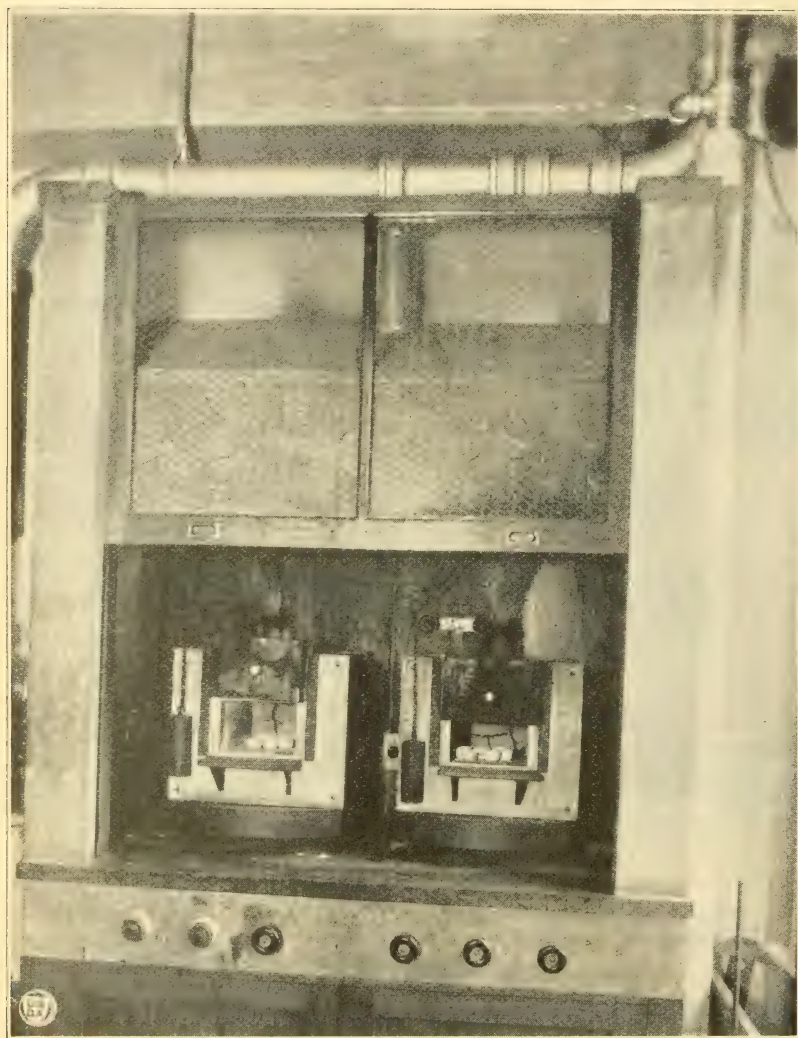


FIG. 30.—Electric furnaces used in making ash determinations on flour.

In cases where extreme accuracy is not needed, where the sample is very small, where time is a factor, or where it is necessary to distinguish with certainty between samples with a medium and those with either a high or low nitrogen content, a great saving in time can be effected without seriously lessening the accuracy of the determination by using whole wheat kernels instead of ground material.



The method is essentially the same as that described by Alway and Bishop.<sup>11</sup> Briefly, about 2 grams of air-dry whole kernels, the exact weight of which is known, are placed in a Kjeldahl flask with 25 cubic centimeters of sulphuric acid, 10 grams of sodium sulphate, 0.7 gram mercuric oxid, and digested and distilled as above. If the data are desired on a water-free basis, a longer time is necessary than that consumed in making tests on a ground sample, as it takes approximately 72 hours to drive all the water out of the whole kernels.



FIG. 31.—Laboratory table showing some of the chemicals used in making protein tests. Apparatus in background used for making color tests on barley and oats. Sieve for washing gluten out of flour to the right.

Nitrogen determinations can, of course, be made on the water-free sample.

#### CRUDE GLUTEN IN FLOUR.

*Qualitative Test*<sup>12</sup> —(Bamihl)—Place a very small quantity (about 1.5 milligrams) of the flour on a microscope slide, add a drop of water containing 0.2 gram of water-soluble eosin in 1 liter, and mix by means of a cover glass, holding the latter at first in such a manner that it is raised slightly above the slide, and taking care that none of the flour escapes from beneath it. Finally, allow the cover glass to rest on the slide and rub it back and

<sup>11</sup> Nebraska Agricultural Experiment Station, 23d Annual Report, 1908-09, p. 21-25: The Determination of Nitrogen in Dried Unground Cereals, by F. J. Alway and E. S. Bishop.

<sup>12</sup> Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists. Revised to November 1, 1919. P. 169.



forth until the gluten has collected into rolls. The operation should be carried out on a white paper so that the formation of gluten rolls can be noted. Wheat flour or other flours containing gluten show by this treatment a copious amount of gluten which absorbs the eosin with avidity assuming a carmine color. Rye and corn flour yield only a trace of gluten; buckwheat flour no appreciable amount. The preparations are best examined with the naked eye, thus gaining an idea of the amount of gluten present. If the flour is coarse, or contains a considerable quantity of bran elements, which is the case

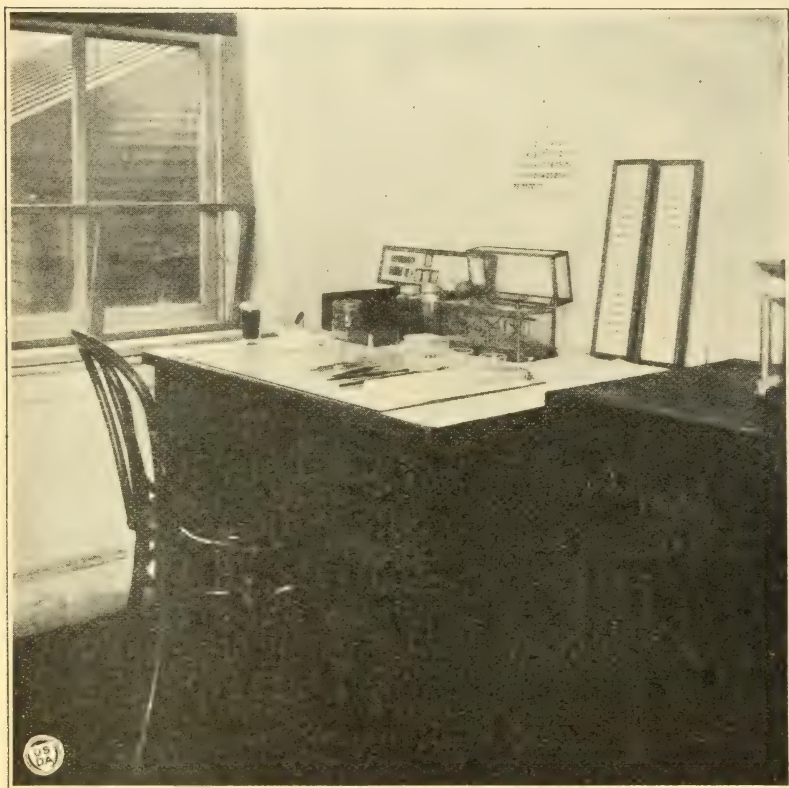


FIG. 32.—A desk with apparatus for qualitative estimation of the quality of the gluten in wheat and flour.

in buckwheat flour and low-grade wheat flour, the test should be made after bolting, as the bran particles and coarse lumps interfere with the formation of gluten rolls. Figure 32 shows the apparatus necessary to make the Bamihl test.

*Quantitative Test.*<sup>13</sup>—Weigh 25 grams of flour and put it into a coffee cup, add tap water equivalent to the water absorption value of the sample, and work into a dough with a spatula or pestle, taking care that none of the material adheres to the vessel. Allow the dough to stand in water at room temperature for one hour. Knead gently under a stream

<sup>13</sup> Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists. Revised to November 1, 1919. P. 169.

of tap water until all the starch and soluble matter is removed. This requires approximately 12 minutes. A piece of fine bolting cloth, stretched over two embroidery hoops or over a large-sized grain sieve, should be held under the dough while washing it. Allow the gluten thus obtained to stand in water for one hour, then press as dry as possible between the hands, roll in a ball in a tared flat-bottom dish and weigh as moist gluten. The transfer to an oven at  $100^{\circ}\text{C}$ . and dry to constant weight, which requires somewhat over 24 hours. Reweigh and calculate for dry gluten. Figure 31 illustrates the apparatus for making quantitative tests for gluten in flour.

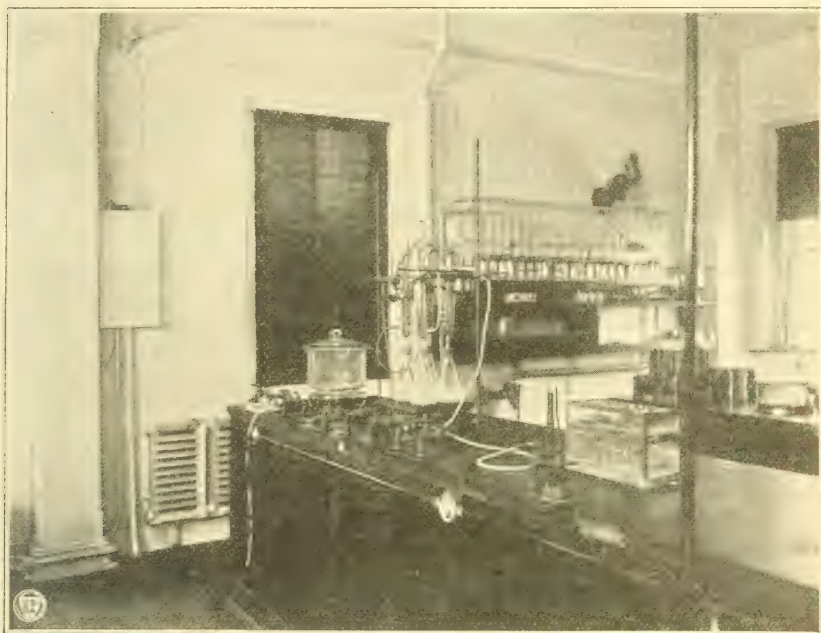


FIG. 33.—Laboratory table showing reflux condensers and electric hotplate for use in making gliadin determination. Fat extraction apparatus rear right of picture.

#### GLIADIN IN FLOUR AND WHEAT.

Put 4 grams of flour and 200 cubic centimeters of 50 per cent ethyl alcohol by volume, specific gravity 0.930 at  $20^{\circ}\text{C}$ ., boiling point  $83^{\circ}$  to  $84^{\circ}\text{C}$ ., into a 500 cubic centimeter Erlenmeyer flask. Connect the flask with a reflux condenser and boil for 3 hours. Then allow it to cool and clarify by centrifuging. Draw off 100 cubic centimeters, representing a 2-gram sample, into a Kjeldahl flask, add 100 cubic centimeters of distilled water, 20 cubic centimeters of sulphuric acid, 10 grams of sodium sulphate, 0.7 gram  $\text{HgO}$ , and determine nitrogen as described. The percentage of nitrogen divided by 2, times 5.7, equals the gliadin.

The following precautions are to be observed: Do not add the sulphuric acid to the alcoholic solution before diluting with water. The addition of water precipitates the gliadin and prevents subsequent frothing, which will take place very easily unless these precautions are observed. The gliadin extraction apparatus is shown in Figure 33.

According to Bailey,<sup>14</sup> the nitrogen extracted by boiling 50 per cent ethyl alcohol from wheat flour is approximately 93 per cent gliadin nitrogen.

**DETERMINATION OF PROTEINS SOLUBLE IN DILUTE SALT SOLUTIONS ( $N \times 5.7 = \text{SALT-SOLUBLE PROTEINS}$ ).<sup>15</sup>**

Weigh 6 grams of the flour into a 200 cubic centimeter flask and introduce exactly 100 cubic centimeters of 5 per cent postassium sulphate solution. Shake at 30 minute intervals for three hours or, better, agitate at moderate speed in a shaker for one hour, let settle 30 minutes, filter and determine the nitrogen in 50 cubic centimeters of the filtrate as directed under "Protein in wheat and flour," page 36.

**COLD WATER SOLUBLE EXTRACT IN FLOUR.<sup>16</sup>**

Weigh 20 grams of the flour into a 500 cubic centimeter Erlenmeyer flask and add gradually 200 cubic centimeters of water at a temperature not higher than 10° C., shake vigorously when about 50 cubic centimeters of water have been added and continue shaking during the addition of the remainder. Allow to stand at 10° C. for 40 minutes, shaking occasionally. Filter through a large, dry, coarse filter paper, returning the first runnings to the filter until a clear filtrate is obtained. Pipette 20 cubic centimeters of the clear filtrate into a tared dish, evaporate to dryness on a steam bath and dry in an oven at 100° C. for periods of 30 minutes to constant weight.

**TOTAL WATER SOLUBLE NITROGEN AND WATER SOLUBLE NON-PROTEIN NITROGEN.<sup>17</sup>**

Place 20 grams of flour into a 500 cubic centimeter Erlenmeyer flask. Add 400 cubic centimeters of distilled water and shake at intervals for 2 hours. At the end of this period clarify by filtration through paper. Determine total nitrogen in 50 cubic centimeters of the filtrate, the equivalent to 2½ grams of material, using the method described above for protein in wheat and flour. This is the total water soluble portion. Pipette 100 cubic centimeters of the flour extract into a 200 cubic centimeter volumetric flask, add 25 cubic centimeters of freshly prepared 0.1 normal sodium hydroxid solution followed by 27 cubic centimeters of 0.1 normal copper sulphate solution. Shake vigorously several times until a water-clear supernatant liquid remains after the precipitate settles. Make to mark, filter, and determine total nitrogen by the Kjeldahl method on 100 cubic centimeters, the equal of 2½ grams of the original sample.

**GLUTENIN (DETERMINATION BY DIFFERENCE).**

Deduct the sum of the potassium sulphate soluble nitrogen and the alcohol-soluble nitrogen from the total nitrogen and multiply the difference by 5.7, taking into consideration the values cited by Bailey and Blish.

<sup>14</sup> Journal of Biological Chemistry, vol. 22, No. 1, p. 345; Concerning the Identity of the Proteins Extracted from Wheat Flour by the Usual Solvents, by C. H. Bailey and M. J. Blish, 1915.

<sup>15</sup> Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists. Revised to November 1, 1919. P. 168, par. 11.

<sup>16</sup> Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists. Revised to November 1, 1919. P. 168, par. 14.

<sup>17</sup> Journal of Biological Chemistry, vol. 33, No. 3, p. 551; A Study of the Nonprotein Nitrogen of Wheat Flour, by M. J. Blish. 1918.



CRUDE FAT.<sup>18</sup>

Wash any of the commercial brands of ether with 2 or 3 successive portions of water, add solid sodium or potassium hydroxid, and let stand until most of the water has been abstracted from the ether. Decant into a dry bottle, add small pieces of carefully cleaned metallic sodium, and let stand until there is no further evolution of hydrogen gas. Keep the ether, thus dehydrated, over metallic sodium in lightly stoppered bottles.

Large quantities of soluble carbohydrates may interfere with the complete extraction of the fat. In such cases extract with water before proceeding with the determination. Extract about 2 grams of material with the anhydrous ether for 16 hours. Dry the extract at the temperature of boiling water for 30 minutes, cool in a desiccator, and weigh; continue, at 30 minute intervals, this alternate drying and weighing to constant weight. For most feeds a period of 1 to 1.5 hours is required.

GASOLINE COLOR VALUE IN FLOUR.<sup>19</sup>

Place 20 grams of the flour in a wide-mouthed, glass-stoppered 120 cubic centimeters bottle and add 100 cubic centimeters of colorless gasoline. Stopper tightly and shake vigorously for 5 minutes. After standing 16 hours, shake again for a few seconds until the flour has been loosened from the bottom of the bottle and thoroughly mixed with the gasoline, then filter immediately on a dry 11 centimeter paper into an Erlenmeyer flask, keeping the funnel covered with a watch glass to prevent evaporation. In order to secure a clear filtrate, a certain quantity of the flour should be allowed to pass over onto the paper and the first portion of the filtrate passed through a second time. It will be found convenient to fit the filter paper to the funnel by means of water. Dry thoroughly either by standing overnight in a well-ventilated place or by heating.

Determine the color value of the clear gasoline solution in a Schreiner or similar colorimeter, using for comparison a 0.005 per cent potassium chromate solution. This solution corresponds to a gasoline number of 1.0 and is conveniently prepared by diluting 10 cubic centimeters of a 0.5 per cent solution to 1 liter. The colorimeter tube, containing the gasoline solution, should first be adjusted so as to read 50 millimeters, then the tube containing the standard chromate solution raised or lowered until the shades of yellow in both tubes match. The reading of the chromate solution, divided by the reading of the gasoline solution, gives the gasoline color value. The color value may be determined also in Nessler tubes, using for comparison potassium chromate solutions of various dilutions prepared from a 0.5 per cent solution and filling the tubes in all cases to the height of 50 millimeters.

The standard is based on the average intensity of a yellow color found in high-grade unbleached spring wheat flour.

Usually the gasoline solution is a true yellow, but sometimes, especially in the case of clear flour, a slight brownish tint is discernible, although this is not sufficient to interfere seriously with the accuracy

<sup>18</sup> Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists. Revised to November 1, 1919. P. 72.

<sup>19</sup> Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists. Revised to November 1, 1919. P. 171.

of the test. Standing for a longer time than prescribed above does not appear to affect the results; in fact, the filtration may be dispensed with entirely if the solution is allowed to settle after the second shaking until perfectly clear, which usually requires at least 24 hours.

If an approximate result is sufficient for the purpose, such may be obtained by carefully pipetting off the clear, supernatant gasoline solution before the second shaking. The results thus obtained are about 10 per cent lower than by the standard method given above.

#### PEKAR COLOR TEST.

Place 10 or 15 grams of the flour on a rectangular glass plate about 12 centimeters long and 8 centimeters wide, and pack on one side in a straight line by means of a flour trier. Treat the same quantity of the standard flour used for comparison in the same manner, so that the straight edges of the two flours are adjacent. Carefully move one of the portions so that it will be in contact with the other, and "slick" both with one stroke of the trier, in such a manner that the thickness of the layer diminishes from about 0.5 centimeter at the middle of the plate to a thin film at the edge, and the line of demarcation between the two flours is distinct. Cut off the edges of the layer with the trier, to form a rectangle. Carefully immerse the plate with the flour in water for one minute. Place in drying oven at 100° C. for about 2 hours or until a rich yellow color develops.

#### TOTAL SUGARS—PREPARATION OF SOLUTION.<sup>20</sup>

Place 12 grams of material in a 300 cubic centimeter graduated flask. If the substance has an acid reaction add 1 to 3 grams of calcium carbonate. Add 150 cubic centimeters of 50 per cent alcohol by volume (carefully neutralized), mix thoroughly, and boil on a steam bath for one hour, using a small funnel in the neck of the flask to condense the vapor. Then cool and allow the mixture to stand several hours; preferably allow it to stand overnight. Make up to volume with 95 per cent alcohol (neutral in reaction), mix thoroughly, allow to settle, transfer 200 cubic centimeters to a beaker with pipette, and evaporate on steam bath to a volume of 20 or 30 cubic centimeters. The presence of a trace of alcohol is not harmful. Do not evaporate the solution to dryness. Transfer the contents of the beaker to a 100 cubic centimeter graduated flask, washing thoroughly with water. Add enough of a saturated solution of neutral lead acetate (60 grams of salt to 100 cubic centimeters of water) to produce a flocculent precipitate, and allow to stand 15 minutes. At this point the solution may safely stand overnight, if desired.

Make up to the mark with distilled water and pass through a folded filter, carefully saving all the filtrate, to which add enough anhydrous sodium carbonate to precipitate all the lead, allow to stand 15 minutes, and pour on an ashless filter. Over 75 cubic centimeters of filtrate should be obtained. Test the filtrate for lead with a small quantity of dry sodium carbonate, and if any precipitation occurs add more anhydrous sodium carbonate and refilter.

<sup>20</sup> Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists. Revised to Nov. 1, 1919. Compiled from p. 94, par. 56; p. 90, par. 52; p. 79, par. 27; p. 78, par. 25; p. 95, par. 58.



## ANALYSIS FOR REDUCING SUGARS—VOLUMETRIC THIOSULPHATE METHOD.

*Reagents: Soxhlet's Modification of Fehling's Solution.*—Prepared by mixing, immediately before use, equal volumes of (a) and (b).

(a) *Copper sulphate solution.*—Dissolve 34.639 grams of copper sulphate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) in water and dilute to 500 cubic centimeters.

(b) *Alkaline tartrate solution.*—Dissolve 173 grams of Rochelle salts and 50 grams of sodium hydroxid in water and dilute to 500 cubic centimeters.

(c) *Standard thiosulphate solution.*—Prepare a solution of sodium thiosulphate containing 10 grams of pure crystals in 1 liter. Weigh accurately about 0.2 gram of pure copper foil and place in a flask of 250 cubic centimeters capacity. Dissolve by warming with 5 cubic centimeters of a mixture of equal volumes of strong nitric acid and water. Dilute to 50 cubic centimeters, boil to expel the red fumes, add 5 cubic centimeters of strong bromin water, and boil until the bromin is completely driven off. Remove from the heat and add a slight excess of strong ammonium hydroxid (about 7 cubic centimeters is required). Again boil until the excess of ammonia is expelled, as shown by a change of color of the liquid, and a partial precipitation. Then add a slight excess of strong acetic acid (3 or 4 cubic centimeters of 80 per cent acid) and boil for a minute. Cool to room temperature and add 10 cubic centimeters of 30 per cent potassium iodid solution. Titrate at once with the thiosulphate solution until the brown tinge has become weak, then add sufficient starch indicator to produce a marked blue coloration. Continue the titration cautiously until the color due to free iodin has entirely vanished. The blue color changes toward the end to a faint lilac. If at this point the thiosulphate be added drop by drop and a little time allowed for complete reaction after each addition, there is no difficulty in determining the end point within a single drop. One cubic centimeter of the thiosulphate solution will be found to correspond to about 0.0005 gram of copper.

(d) *Starch indicator.*—Mix about 0.5 gram of finely powdered potato starch with cold water to a thin paste; pour into about 100 cubic centimeters of boiling water, stirring constantly, and discontinue heating immediately after the paste is added.

*Determination.*—Transfer 25 cubic centimeters each of the copper sulphate and alkaline tartrate solutions to a 400 cubic centimeter beaker of alkali-resistant glass and add 25 cubic centimeters of reducing sugar solution, or, if a smaller volume of sugar solution is used, add water to make the final volume 100 cubic centimeters. Heat the beaker upon an asbestos gauze over a Bunsen burner, regulate the flame so that boiling begins in 4 minutes and continue the boiling for exactly 2 minutes. (It is important that these directions be strictly observed and, in order to regulate the burner for this purpose, it is advisable to make preliminary tests, using 50 cubic centimeters of the reagent and 50 cubic centimeters of water before proceeding with the actual determination.) Keep the beaker covered with a watch glass during the heating. Filter the cuprous oxid at once on an asbestos mat in a porcelain Gooch crucible, using suction. Wash the cuprous oxid thoroughly with water at a temperature of about  $60^\circ\text{C}$ . Cover the Gooch with a watch glass and dissolve the oxid by means of 5 cubic centimeters of warm nitric acid (1 to 1) poured under the watch glass with a pipette. Catch the filtrate in a 250 cubic centimeter flask, wash the watch glass and Gooch free of copper, using about 50 cubic centimeters of water. Boil to expel red fumes, add 5 cubic centimeters of bromin water, boil off the bromin and proceed exactly as directed under "Standard thiosulphate solution." Calculate the milligrams of copper reduced and from Table 3 the weight of invert sugar equivalent to the weight of copper found. Divide the result by 2 and multiply by 100 to obtain the percentage of sugar as invert.

## SUCROSE.

In a 100 cubic centimeter graduated flask place 50 cubic centimeters of the same filtrate, add a small piece of litmus paper, and neutralize with acetic acid; then add 5 cubic centimeters of concen-



trated hydrochloric acid, and let stand overnight for inversion. (Standing 48 hours does not apparently affect the results.) Then pour the inverted solution into a 400 cubic centimeter beaker and neutralize with anhydrous sodium carbonate; return it to the 100 cubic centimeter flask and make up to the mark. Filter, if necessary, and use 50 cubic centimeters for the determination of total sugars as invert, as directed above.

The amount of copper obtained in either the reducing or the total sugar determinations represents the sugar contained in 2 grams of the material. Therefore the weights of the invert sugar when divided by 2 and multiplied by 100 give the respective percentages of sugar as invert. Subtract the percentage of reducing sugars before inversion from the percentages of total sugar after inversion, both calculated as invert, and the difference multiplied by 0.95 gives the percentage of sucrose. Since the insoluble matter of the material to be analyzed occupies some space in the flask as originally made up, it is necessary to correct for this volume. Results of a large number of determinations on various materials have shown the average volume of 12 grams of material to be 9 cubic centimeters; therefore the correction factor for 12 grams in 300 cubic centimeters is 0.97 and the percentage figures for reducing sugar and sucrose are to be multiplied by this factor to obtain the true amounts.<sup>21</sup>

The number of milligrams of copper reduced by a given amount of reducing sugar differs when sucrose is present and when it is absent. In the tables the absence of sucrose is assumed, except in the two columns under invert sugar, where one for mixtures of invert sugar and sucrose containing 0.4 gram of total sugar in 50 cubic centimeters of solution, and one for invert sugar and sucrose when the 50 cubic centimeters of solution contains 2 grams of total sugar are given, in addition to the column for invert sugar alone.

#### STARCH-DIASTASE METHOD WITH SUBSEQUENT ACID HYDROLYSIS.<sup>22</sup>

**Reagents.**—*Malt Extract.*—Digest 10 grams of fresh, finely ground malt with 200 cubic centimeters of water for 2 or 3 hours at ordinary temperature and filter. Determine the amount of dextrose in a given quantity of the filtrate after boiling with acid, etc., as in the starch determination, and make the proper correction in the subsequent determination.

**Determination.**—Extract 5 grams of the substance (ground to an impalpable powder and representing 4 or 5 grams of the dry material) on a hardened filter with 5 successive portions of 10 cubic centimeters of ether; wash with 150 cubic centimeters of 50 per cent alcohol (10 per cent alcohol has not been found satisfactory, because it will extract some soluble starch). Place the residue in a beaker with 50 cubic centimeters of water, immerse the beaker in boiling water, and stir constantly for 15 minutes or until all the starch is gelatinized; cool to 55° C., add 20 cubic centimeters of malt extract and heat gradually for an hour, raising the temperature to 72° C. in this time. Heat again to boiling for a few minutes, cool to 55° C., add 20 cubic centimeters of malt extract and maintain at this temperature for an hour or until the residue treated with iodine shows no blue color upon microscopic examination. Cool, make up directly to

<sup>21</sup> U. S. Department of Agriculture, Bureau of Chemistry Circular 71, Extraction of Grains and Cattle Foods for the Determination of Sugars, by A. H. Bryan, A. Given, and M. N. Straughn, p. 3, 1911.

<sup>22</sup> Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists. Revised to November 1, 1919, p. 95, par. 60 and 61.

250 cubic centimeters and filter. Place 200 cubic centimeters of the filtrate in a flask with 20 cubic centimeters of hydrochloric acid (specific gravity 1.125); connect with a reflux condenser and heat in a boiling water bath for 2.5 hours. Cool, nearly neutralize with sodium hydroxid solution, finish the neutralization with sodium carbonate solution and make up to 500 cubic centimeters. Mix the solution well, pour through a dry filter, and determine the dextrose in an aliquot as described under reducing sugars. Conduct a blank determination upon the same volume of the malt extract as used upon the sample and correct the weight of reduced copper accordingly. The weight of the dextrose obtained multiplied by 0.90 gives the weight of starch.

#### CRUDE FIBER.<sup>23</sup>

*Reagents.*—(a) *Dilute sulphuric acid solution.*—Contains exactly 1.25 grams of sulphuric acid in 100 cubic centimeters as determined by titration.

(b) *Dilute sodium hydroxid solution.*—Contains exactly 1.25 grams of sodium hydroxid in 100 cubic centimeters as determined by titration. This solution should be free, or practically free, from sodium carbonate.

#### Reagents—

*Determination.*—Extract a quantity of the substance, representing about 2 grams of the dry material, with ordinary ether, or use the residue from the determination of the ether extract. Put this residue in a 500 cubic centimeter flask and add 200 cubic centimeters of the boiling dilute sulphuric acid solution; connect the flask with a reflux condenser, the tube of which passes only a short distance beyond the rubber stopper into the flask, or simply cover a tall conical flask, which is well suited for this determination, with a watch glass or short-stemmed funnel, boil at once, and continue boiling gently for 30 minutes. Filter through linen, and wash with boiling water until the washings are no longer acid; rinse the substance back into the flask with 200 cubic centimeters of boiling dilute sodium hydroxide solution; boil at once, and continue boiling gently for 30 minutes as directed above for the treatment with acid. Filter at once rapidly upon a linen filter until the washings are neutral. Transfer to a weighed Gooch crucible, and dry to constant weight at 110° C. Weigh, incinerate completely, and weigh again. The loss in weight is considered crude fiber.

#### ERGOT.

Ergot, a fungous growth containing a poisonous alkaloid, sometimes develops in rye and, less often, in wheat. Under the microscope it appears as a fine network of mostly colorless parenchyma cells, containing globules of fat. Some of the cells are circular, others considerably elongated, and some contain a deep-brown coloring matter which, with ammonia, becomes violet red, or with acid becomes red. Occasionally the cell walls appear of a dark color. If flour containing ergot be treated with a very dilute solution of anilin violet, the stain will be practically absorbed by the damaged particles of the grain and resisted by the normal granules. A hot alcoholic extract of flour containing ergot is colored red when treated with dilute sulphuric acid.

<sup>23</sup> Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists. Revised to November 1, 1919, p. 97.

## REFERENCE TABLES.

TABLE 1.—Quantity of water required in tempering wheats of various moisture contents.

Moisture content of wheat.	Quantity of water required to raise moisture content of wheat to—				Moisture content of wheat.	Quantity of water required to raise moisture content of wheat to—			
	14 per cent.		15 per cent.			14 per cent.		15 per cent.	
	Per 1,500 grams wheat.	Per 100 grams wheat.	Per 1,500 grams wheat.	Per 100 grams wheat.		Per 1,500 grams wheat.	Per 100 grams wheat.	Per 1,500 grams wheat.	Per 100 grams wheat.
<i>Per cent.</i>	<i>c. c.</i>	<i>c. c.</i>	<i>c. c.</i>	<i>c. c.</i>	<i>Per cent.</i>	<i>c. c.</i>	<i>c. c.</i>	<i>c. c.</i>	<i>c. c.</i>
6.0	139.5	9.3023	158.8	10.5882	10.5	61.0	4.0697	79.4	5.2942
6.1	137.7	9.1860	157.1	10.4706	10.6	59.3	3.9534	77.6	5.1765
6.2	136.0	9.0697	155.3	10.3529	10.7	57.6	3.8372	75.9	5.0589
6.3	134.3	8.9534	153.5	10.2353	10.8	55.8	3.7209	74.1	4.9412
6.4	132.6	8.8372	151.8	10.1177	10.9	54.1	3.6046	72.4	4.8235
6.5	130.8	8.7209	150.0	10.0000	11.0	52.3	3.4883	70.6	4.7059
6.6	129.1	8.6046	148.2	9.8823	11.1	50.6	3.3720	68.8	4.5883
6.7	127.3	8.4883	146.5	9.7647	11.2	48.8	3.2558	67.1	4.4706
6.8	125.6	8.3720	144.7	9.6470	11.3	47.1	3.1395	65.3	4.3530
6.9	123.8	8.2558	142.9	9.5295	11.4	45.3	3.0232	63.5	4.2353
7.0	122.1	8.1395	141.2	9.4118	11.5	43.6	2.9069	61.8	4.1177
7.1	120.3	8.0232	139.4	9.2924	11.6	41.9	2.7907	60.0	4.0000
7.2	118.6	7.9069	137.6	9.1765	11.7	40.1	2.6744	58.2	3.8824
7.3	116.9	7.7907	135.9	9.0589	11.8	38.4	2.5581	56.5	3.7647
7.4	115.1	7.6744	134.1	8.9412	11.9	36.6	2.4418	54.7	3.6471
7.5	113.4	7.5581	132.4	8.8236	12.0	34.9	2.3256	52.9	3.5294
7.6	111.6	7.4418	130.6	8.7059	12.1	33.1	2.2093	51.2	3.4118
7.7	109.9	7.3256	128.8	8.5883	12.2	31.4	2.0930	49.4	3.2941
7.8	108.1	7.2093	127.1	8.4706	12.3	29.7	1.9767	47.6	3.1765
7.9	106.4	7.0930	125.3	8.3530	12.4	27.9	1.8604	45.9	3.0588
8.0	104.7	6.9767	123.5	8.2353	12.5	26.2	1.7441	44.1	2.9412
8.1	102.9	6.8604	121.8	8.1177	12.6	24.4	1.6279	42.4	2.8235
8.2	101.2	6.7441	120.0	8.0000	12.7	22.7	1.5116	40.6	2.7059
8.3	99.4	6.6279	118.2	7.8824	12.8	20.9	1.3953	38.8	2.5882
8.4	97.7	6.5116	116.5	7.7648	12.9	19.2	1.2790	37.1	2.4706
8.5	95.9	6.3953	114.7	7.6471	13.0	17.4	1.1628	35.3	2.3529
8.6	94.2	6.2790	112.9	7.5294	13.1	15.7	1.0465	33.5	2.2353
8.7	92.4	6.1628	111.2	7.4118	13.2	14.0	.9302	31.8	2.1176
8.8	90.7	6.0465	109.4	7.2941	13.3	12.2	.8139	30.0	2.0000
8.9	89.0	5.9302	107.6	7.1765	13.4	10.5	.6976	28.2	1.8823
9.0	87.2	5.8139	105.9	7.0588	13.5	8.7	.5814	26.5	1.7647
9.1	85.5	5.6976	104.1	6.9412	13.6	7.0	.4651	24.7	1.6471
9.2	83.7	5.5813	102.4	6.8235	13.7	5.2	.3488	22.9	1.5294
9.3	82.0	5.4651	100.6	6.7059	13.8	3.5	.2325	21.2	1.4117
9.4	80.2	5.3488	98.8	6.5882	13.9	1.7	.1163	19.4	1.2941
9.5	78.5	5.2325	97.1	6.4706	14.0	.0	.0000	17.6	1.1765
9.6	76.7	5.1162	95.3	6.3529	14.1	-----	-----	15.9	1.0588
9.7	75.0	5.0000	93.5	6.2353	14.2	-----	-----	14.1	.9412
9.8	73.3	4.8837	91.8	6.1175	14.3	-----	-----	12.4	.8235
9.9	71.5	4.7674	90.0	6.0000	14.4	-----	-----	10.6	.7059
10.0	69.8	4.6511	88.2	5.8823	14.5	-----	-----	8.8	.5882
10.1	68.0	4.5349	86.5	5.7647	14.6	-----	-----	7.1	.4706
10.2	66.3	4.4186	84.7	5.6470	14.7	-----	-----	5.3	.3529
10.3	64.5	4.3023	82.9	5.5294	14.8	-----	-----	3.5	.2353
10.4	62.8	4.1860	81.2	5.4118	14.9	-----	-----	1.8	.1176



TABLE 2.—*Figures for converting total nitrogen percentages of wheat and flour into crude protein percentages.*<sup>1</sup>

Nitro- gen.	Protein.	Nitro- gen.	Protein.	Nitro- gen.	Protein.	Nitro- gen.	Protein.
<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
1.00....	5.70						
1.01....	5.76	1.51....	8.61	2.01....	11.46	2.51....	14.31
1.02....	5.81	1.52....	8.66	2.02....	11.51	2.52....	14.36
1.03....	5.87	1.53....	8.72	2.03....	11.57	2.53....	14.42
1.04....	5.93	1.54....	8.78	2.04....	11.63	2.54....	14.48
1.05....	5.99	1.55....	8.83	2.05....	11.68	2.55....	14.53
1.06....	6.04	1.56....	8.89	2.06....	11.74	2.56....	14.59
1.07....	6.10	1.57....	8.95	2.07....	11.80	2.57....	14.65
1.08....	6.16	1.58....	9.01	2.08....	11.86	2.58....	14.71
1.09....	6.21	1.59....	9.06	2.09....	11.91	2.59....	14.76
1.10....	6.27	1.60....	9.12	2.10....	11.97	2.60....	14.82
1.11....	6.33	1.61....	9.18	2.11....	12.03	2.61....	14.88
1.12....	6.38	1.62....	9.23	2.12....	12.08	2.62....	14.93
1.13....	6.44	1.63....	9.29	2.13....	12.14	2.63....	14.99
1.14....	6.50	1.64....	9.35	2.14....	12.20	2.64....	15.05
1.15....	6.55	1.65....	9.40	2.15....	12.25	2.65....	15.10
1.16....	6.61	1.66....	9.46	2.16....	12.31	2.66....	15.16
1.17....	6.67	1.67....	9.52	2.17....	12.37	2.67....	15.22
1.18....	6.73	1.68....	9.58	2.18....	12.43	2.68....	15.28
1.19....	6.78	1.69....	9.63	2.19....	12.48	2.69....	15.33
1.20....	6.84	1.70....	9.69	2.20....	12.54	2.70....	15.39
1.21....	6.90	1.71....	9.75	2.21....	12.60	2.71....	15.45
1.22....	6.95	1.72....	9.80	2.22....	12.65	2.72....	15.50
1.23....	7.01	1.73....	9.86	2.23....	12.71	2.73....	15.56
1.24....	7.07	1.74....	9.92	2.24....	12.77	2.74....	15.62
1.25....	7.12	1.75....	9.97	2.25....	12.82	2.75....	15.67
1.26....	7.18	1.76....	10.03	2.26....	12.88	2.76....	15.73
1.27....	7.24	1.77....	10.09	2.27....	12.94	2.77....	15.79
1.28....	7.30	1.78....	10.15	2.28....	13.00	2.78....	15.85
1.29....	7.35	1.79....	10.20	2.29....	13.05	2.79....	15.90
1.30....	7.41	1.80....	10.26	2.30....	13.11	2.80....	15.96
1.31....	7.47	1.81....	10.32	2.31....	13.17	2.81....	16.02
1.32....	7.52	1.82....	10.37	2.32....	13.22	2.82....	16.07
1.33....	7.58	1.83....	10.43	2.33....	13.28	2.83....	16.13
1.34....	7.64	1.84....	10.49	2.34....	13.34	2.84....	16.19
1.35....	7.69	1.85....	10.54	2.35....	13.39	2.85....	16.24
1.36....	7.75	1.86....	10.60	2.36....	13.45	2.86....	16.30
1.37....	7.81	1.87....	10.66	2.37....	13.51	2.87....	16.36
1.38....	7.87	1.88....	10.72	2.38....	13.57	2.88....	16.42
1.39....	7.92	1.89....	10.77	2.39....	13.62	2.89....	16.47
1.40....	7.98	1.90....	10.83	2.40....	13.68	2.90....	16.53
1.41....	8.04	1.91....	10.89	2.41....	13.74	2.91....	16.59
1.42....	8.09	1.92....	10.94	2.42....	13.79	2.92....	16.64
1.43....	8.15	1.93....	11.00	2.43....	13.85	2.93....	16.70
1.44....	8.21	1.94....	11.06	2.44....	13.91	2.94....	16.76
1.45....	8.26	1.95....	11.11	2.45....	13.96	2.95....	16.81
1.46....	8.32	1.96....	11.17	2.46....	14.02	2.96....	16.87
1.47....	8.38	1.97....	11.23	2.47....	14.08	2.97....	16.93
1.48....	8.44	1.98....	11.29	2.48....	14.14	2.98....	16.99
1.49....	8.49	1.99....	11.34	2.49....	14.19	2.99....	17.04
1.50....	8.55	2.00....	11.40	2.50....	14.25	3.00....	17.10

<sup>1</sup> Conversion factor 5.7.

TABLE 2.—*Figures for converting total nitrogen percentages of wheat and flour into crude protein percentages*<sup>1</sup>—Continued.

Nitro- gen.	Protein.	Nitro- gen.	Protein.	Nitro- gen.	Protein.	Nitro- gen.	Protein.
<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
3.01....	17.16	3.51....	20.01	4.01....	22.86	4.51....	25.71
3.02....	17.21	3.52....	20.06	4.02....	22.91	4.52....	25.76
3.03....	17.27	3.53....	20.12	4.03....	22.97	4.53....	25.82
3.04....	17.33	3.54....	20.18	4.04....	23.03	4.54....	25.88
3.05....	17.38	3.55....	20.23	4.05....	23.08	4.55....	25.93
3.06....	17.44	3.56....	20.29	4.06....	23.14	4.56....	25.99
3.07....	17.50	3.57....	20.35	4.07....	23.20	4.57....	26.05
3.08....	17.56	3.58....	20.41	4.08....	23.26	4.58....	26.11
3.09....	17.61	3.59....	20.46	4.09....	23.31	4.59....	26.16
3.10....	17.67	3.60....	20.52	4.10....	23.37	4.60....	26.22
3.11....	17.73	3.61....	20.58	4.11....	23.43	4.61....	26.28
3.12....	17.78	3.62....	20.63	4.12....	23.48	4.62....	26.33
3.13....	17.84	3.63....	20.69	4.13....	23.54	4.63....	26.39
3.14....	17.90	3.64....	20.75	4.14....	23.60	4.64....	26.45
3.15....	17.95	3.65....	20.80	4.15....	23.65	4.65....	26.50
3.16....	18.01	3.66....	20.86	4.16....	23.71	4.66....	26.56
3.17....	18.07	3.67....	20.92	4.17....	23.77	4.67....	26.62
3.18....	18.13	3.68....	20.98	4.18....	23.83	4.68....	26.68
3.19....	18.18	3.69....	21.03	4.19....	23.88	4.69....	26.73
3.20....	18.24	3.70....	21.09	4.20....	23.94	4.70....	26.79
3.21....	18.30	3.71....	21.15	4.21....	24.00	4.71....	26.85
3.22....	18.35	3.72....	21.20	4.22....	24.05	4.72....	26.90
3.23....	18.41	3.73....	21.26	4.23....	24.11	4.73....	26.96
3.24....	18.47	3.74....	21.32	4.24....	24.17	4.74....	27.02
3.25....	18.52	3.75....	21.37	4.25....	24.22	4.75....	27.07
3.26....	18.58	3.76....	21.43	4.26....	24.28	4.76....	27.13
3.27....	18.64	3.77....	21.49	4.27....	24.34	4.77....	27.19
3.28....	18.70	3.78....	21.55	4.28....	24.40	4.78....	27.25
3.29....	18.75	3.79....	21.60	4.29....	24.45	4.79....	27.30
3.30....	18.81	3.80....	21.66	4.30....	24.51	4.80....	27.36
3.31....	18.87	3.81....	21.72	4.31....	24.57	4.81....	27.42
3.32....	18.92	3.82....	21.77	4.32....	24.62	4.82....	27.47
3.33....	18.98	3.83....	21.83	4.33....	24.68	4.83....	27.53
3.34....	19.04	3.84....	21.89	4.34....	24.74	4.84....	27.59
3.35....	19.09	3.85....	21.94	4.35....	24.79	4.85....	27.64
3.36....	19.15	3.86....	22.00	4.36....	24.85	4.86....	27.70
3.37....	19.21	3.87....	22.06	4.37....	24.91	4.87....	27.76
3.38....	19.27	3.88....	22.12	4.38....	24.97	4.88....	27.82
3.39....	19.32	3.89....	22.17	4.39....	25.02	4.89....	27.87
3.40....	19.38	3.90....	22.23	4.40....	25.08	4.90....	27.93
3.41....	19.44	3.91....	22.29	4.41....	25.14	4.91....	27.99
3.42....	19.49	3.92....	22.34	4.42....	25.19	4.92....	28.04
3.43....	19.55	3.93....	22.40	4.43....	25.25	4.93....	28.10
3.44....	19.61	3.94....	22.46	4.44....	25.31	4.94....	28.16
3.45....	19.66	3.95....	22.51	4.45....	25.36	4.95....	28.21
3.46....	19.72	3.96....	22.57	4.46....	25.42	4.96....	28.27
3.47....	19.78	3.97....	22.63	4.47....	25.48	4.97....	28.33
3.48....	19.84	3.98....	22.69	4.48....	25.54	4.98....	28.39
3.49....	19.89	3.99....	22.74	4.49....	25.59	4.99....	28.44
3.50....	19.95	4.00....	22.80	4.50....	25.65	5.00....	28.50

<sup>1</sup> Conversion factor 5.7.

TABLE 3.—*Munson and Walker's table for calculating invert sugar alone and invert sugar in the presence of sucrose (0.4 gram and 2 grams total sugar).<sup>1</sup>*

(Expressed in milligrams.)

Cuprous oxid (Cu <sub>2</sub> O).	Copper (Cu).	Invert sugar.	Invert sugar and sucrose.		Cuprous oxid (Cu <sub>2</sub> O).	Copper (Cu).	Invert sugar.	Invert sugar and sucrose.	
			0.4 gram total sugar.	2 grams total sugar.				0.4 gram total sugar.	2 grams total sugar.
10	8.9	4.5	1.6	.....	65	57.7	29.1	26.6	20.3
11	9.8	5.0	2.1	.....	66	58.6	29.5	27.1	20.8
12	10.7	5.4	2.5	.....	67	59.5	30.0	27.5	21.2
13	11.5	5.8	3.0	.....	68	60.4	30.4	28.0	21.7
14	12.4	6.3	3.4	.....	69	61.3	30.9	28.5	22.2
15	13.3	6.7	3.9	.....	70	62.2	31.3	28.9	22.6
16	14.2	7.2	4.3	.....	71	63.1	31.8	29.4	23.1
17	15.1	7.6	4.8	.....	72	64.0	32.3	29.8	23.5
18	16.0	8.1	5.2	.....	73	64.8	32.7	30.3	24.0
19	16.9	8.5	5.7	.....	74	65.7	33.2	30.8	24.5
20	17.8	8.9	6.1	.....	75	66.6	33.6	31.2	24.9
21	18.7	9.4	6.6	.....	76	67.5	34.1	31.7	25.4
22	19.5	9.8	7.0	.....	77	68.4	34.5	32.1	25.9
23	20.4	10.3	7.5	.....	78	69.3	35.0	32.6	26.3
24	21.3	10.7	7.9	.....	79	70.2	35.4	33.1	26.8
25	22.2	11.2	8.4	.....	80	71.1	35.9	33.5	27.3
26	23.1	11.6	8.8	.....	81	71.9	36.3	34.0	27.7
27	24.0	12.0	9.3	.....	82	72.8	36.8	34.5	28.2
28	24.9	12.5	9.7	.....	83	73.7	37.3	34.9	28.6
29	25.8	12.9	10.2	.....	84	74.6	37.7	35.4	29.1
30	26.6	13.4	10.7	4.3	85	75.5	38.2	35.8	29.6
31	27.5	13.8	11.1	4.7	86	76.4	38.6	36.3	30.0
32	28.4	14.3	11.6	5.2	87	77.3	39.1	36.8	30.5
33	29.3	14.7	12.0	5.6	88	78.2	39.5	37.2	31.0
34	30.2	15.2	12.5	6.1	89	79.1	40.0	37.7	31.4
35	31.1	15.6	12.9	6.5	90	79.9	40.4	38.2	31.9
36	32.0	16.1	13.4	7.0	91	80.8	40.9	38.6	32.4
37	32.9	16.5	13.8	7.4	92	81.7	41.4	39.1	32.8
38	33.8	16.9	14.3	7.9	93	82.6	41.8	39.6	33.3
39	34.6	17.4	14.7	8.4	94	83.5	42.3	40.0	33.8
40	35.5	17.8	15.2	8.8	95	84.4	42.7	40.5	34.2
41	36.4	18.3	15.6	9.3	96	85.3	43.2	41.0	34.7
42	37.3	18.7	16.1	9.7	97	86.2	43.7	41.4	35.2
43	38.2	19.2	16.6	10.2	98	87.1	44.1	41.9	35.6
44	39.1	19.6	17.0	10.7	99	87.9	44.6	42.4	36.1
45	40.0	20.1	17.5	11.1	100	88.8	45.0	42.8	36.6
46	40.9	20.5	17.9	11.6	101	89.7	45.5	43.3	37.0
47	41.7	21.0	18.4	12.0	102	90.6	46.0	43.8	37.5
48	42.6	21.4	18.8	12.5	103	91.5	46.4	44.2	38.0
49	43.5	21.9	19.3	12.9	104	92.4	46.9	44.7	38.5
50	44.4	22.3	19.7	13.4	105	93.3	47.3	45.2	38.9
51	45.3	22.8	20.2	13.9	106	94.2	47.8	45.6	39.4
52	46.2	23.2	20.7	14.3	107	95.0	48.3	46.1	39.9
53	47.1	23.7	21.1	14.8	108	95.9	48.7	46.6	40.3
54	48.0	24.1	21.6	15.2	109	96.8	49.2	47.0	40.8
55	48.9	24.6	22.0	15.7	110	97.7	49.6	47.5	41.3
56	49.7	25.0	22.5	16.2	111	98.6	50.1	48.0	41.7
57	50.6	25.5	22.9	16.6	112	99.5	50.6	48.4	42.2
58	51.5	25.9	23.4	17.1	113	100.4	51.0	48.9	42.7
59	52.4	26.4	23.9	17.5	114	101.3	51.5	49.4	43.2
60	53.3	26.8	24.3	18.0	115	102.2	51.9	49.8	43.6
61	54.2	27.3	24.8	18.5	116	103.0	52.4	50.3	44.1
62	55.1	27.7	25.2	18.9	117	103.9	52.9	50.8	44.6
63	56.0	28.2	25.7	19.4	118	104.8	53.3	51.2	45.0
64	56.8	28.6	26.2	19.8	119	105.7	53.8	51.7	45.5

<sup>1</sup> Taken from Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists, revised to November 1, 1919, pp. 321 to 325, inclusive.



TABLE 3.—*Manson and Walker's table for calculating invert sugar alone and invert sugar in the presence of sucrose (0.4 gram and 2 grams total sugar)—Continued.*

(Expressed in milligrams.)

Cuprous oxid (Cu <sub>2</sub> O).	Copper (Cu).	Invert sugar.	Invert sugar and sucrose.		Cuprous oxid (Cu <sub>2</sub> O).	Copper (Cu).	Invert sugar.	Invert sugar and sucrose.	
			0.4 gram total sugar.	2 grams total sugar.				0.4 gram total sugar.	2 grams total sugar.
120	106.6	54.3	52.2	46.0	160	142.1	73.0	71.2	65.0
121	107.5	54.7	52.7	46.5	161	143.0	73.4	71.6	65.5
122	108.4	55.2	53.1	46.9	162	143.9	73.9	72.1	66.0
123	109.3	55.7	53.6	47.4	163	144.8	74.4	72.6	66.5
124	110.1	56.1	54.1	47.9	164	145.7	74.9	73.1	66.9
125	111.0	56.6	54.5	48.3	165	146.6	75.3	73.6	67.4
126	111.9	57.0	55.0	48.8	166	147.5	75.8	74.0	67.9
127	112.8	57.5	55.5	49.3	167	148.3	76.3	74.5	68.4
128	113.7	58.0	55.9	49.8	168	149.2	76.8	75.0	68.9
129	114.6	58.4	56.4	50.2	169	150.1	77.2	75.5	69.3
130	115.5	58.9	56.9	50.7	170	151.0	77.7	76.0	69.8
131	116.4	59.4	57.4	51.2	171	151.9	78.2	76.4	70.3
132	117.3	59.8	57.8	51.7	172	152.8	78.7	76.9	70.8
133	118.1	60.3	58.3	52.1	173	153.7	79.1	77.4	71.3
134	119.0	60.8	58.8	52.6	174	154.6	79.6	77.9	71.7
135	119.9	61.2	59.3	53.1	175	155.5	80.1	78.4	72.2
136	120.8	61.7	59.7	53.6	176	156.3	80.6	78.8	72.7
137	121.7	62.2	60.2	54.0	177	157.2	81.0	79.3	73.2
138	122.6	62.6	60.7	54.5	178	158.1	81.5	79.8	73.7
139	123.5	63.1	61.2	55.0	179	159.0	82.0	80.3	74.2
140	124.4	63.6	61.6	55.5	180	159.9	82.5	80.8	74.6
141	125.2	64.0	62.1	55.9	181	160.8	82.9	81.3	75.1
142	126.1	64.5	62.6	56.4	182	161.7	83.4	81.7	75.6
143	127.0	65.0	63.1	56.9	183	162.6	83.9	82.2	76.1
144	127.9	65.4	63.5	57.4	184	163.4	84.4	82.7	76.6
145	128.8	65.9	64.0	57.8	185	164.3	84.9	83.2	77.1
146	129.7	66.4	64.5	58.3	186	165.2	85.3	83.7	77.6
147	130.6	66.9	65.0	58.8	187	166.1	85.8	84.2	78.0
148	131.5	67.3	65.4	59.3	188	167.0	86.3	84.6	78.5
149	132.4	67.8	65.9	59.7	189	167.9	86.8	85.1	79.0
150	133.2	68.3	66.4	60.2	190	168.8	87.2	85.6	79.5
151	134.1	68.7	66.9	60.7	191	169.7	87.7	86.1	80.0
152	135.0	69.2	67.3	61.2	192	170.5	88.2	86.6	80.5
153	135.9	69.7	67.8	61.7	193	171.4	88.7	87.1	81.0
154	136.8	70.1	68.3	62.1	194	172.3	89.2	87.6	81.4
155	137.7	70.6	68.8	62.6	195	173.2	89.6	88.0	81.9
156	138.6	71.1	69.2	63.1	196	174.1	90.1	88.5	82.4
157	139.5	71.6	69.7	63.6	197	175.0	90.6	89.0	82.9
158	140.3	72.0	70.2	64.1	198	175.9	91.1	89.5	83.4
159	141.2	72.5	70.7	64.5	199	176.8	91.6	90.0	83.9

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